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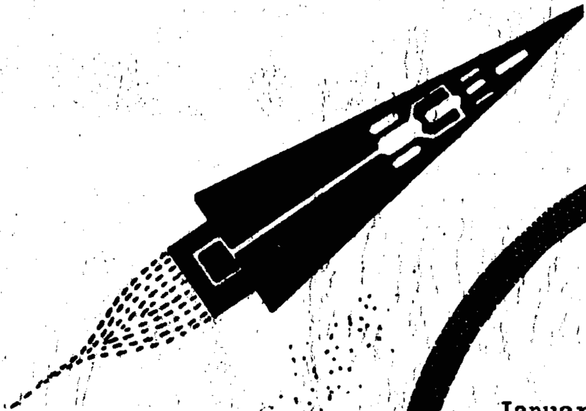

FINAL REPORT

Covering the Period

January 22, 1964 to January 22, 1965

CONTAMINATION EXPOSURES OF COLUMBIUM AND Cb-1Zr ALLOY SPECIMENS

Contract NAS 3-4169

THE NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

MISSILE and SPACE DIVISION

GENERAL ELECTRIC

CINCINNATI, OHIO

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Re-entry Systems Department
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General Electric Company
Cincinnati, Ohio

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Contamination Exposures of Columbium and Cb-1Zr Alloy Specimens

By

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and

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ABSTRACT

This experimental program was conducted to determine the oxidation rate of columbium and columbium-zirconium alloys under conditions of low pressures and high temperatures.

A total of nine exposures of small specimens were conducted, with oxygen pressures at levels ranging from about 1×10^{-7} to about 5×10^{-5} torr, and with specimen temperature levels of 980 and 1100 deg C.

The oxidation or gettering rates were determined by the method of measuring the pressure drop across a known flow conductance, from which the oxygen gas mass flow rates (equal to the gettering rates) were computed. These gettering rates plus predictions of oxygen pressures over the specimens, were used by the NASA Lewis Research Center in calculating sticking factors for oxygen on the metal surfaces.

The report includes ion gage calibrations for oxygen and nitrogen, and analyses of pressure distribution within the system.

9 June 1967

CONTAMINATION EXPOSURES OF COLUMBIUM AND Cb-1Zr ALLOY SPECIMENS

NASA CONTRACT NAS 3-4169

1. INTRODUCTION AND SUMMARY

The program described by this report was conducted to determine the oxidation rate, or gettering rate for oxygen, of columbium and Cb-1Zr alloy specimens under conditions of low pressures and high temperatures.

As indicated below, nine exposures were conducted during the program, with the pressure ranging from 10^{-5} torr to 5×10^{-7} torr, and the temperature mainly at 980°C and 1100°C.

Exposure No.	Specimen No.	Approximate Pressure Level, Torr	Temperature, °C
1	Cb-1Zr—34	10^{-6}	980
2	Cb — 3	Mid 10^{-5}	927
3	Cb-1Zr—35	10^{-5}	1100
4	Cb — 1	10^{-6}	980
5	Cb-1Zr—48	10^{-6}	980
6	Cb-1Zr—50	*	*
7	Cb-1Zr—36	*	*
8	Cb-1Zr—37	*	*
9	Cb —52	*	*

* A series of pressures and temperatures for each exposure.

The gettering rates were established by the method of measuring the pressure drop across a known flow conductance, from which a gas flow rate (equal to the gettering rate) can be computed.

In addition to the nine exposures, the program included five tasks which were performed to improve the accuracy of measurements, or the reliability of the apparatus. These tasks were:

(1) Establishment of the flow conductance experimentally. Previous work had used values for the flow conductance which had been established by measuring the physical dimensions of the flow passage and computing the conductance theoretically.

(2) Installation of new ion gages with thoria-coated iridium filaments. This new type filament has been found to eliminate the problem of conversion of O_2 to CO in a reaction which takes place on tungsten filaments, and to eliminate the degradation of the filament which occurs when tungsten filaments are exposed to oxygen for long periods.

(3) Use of a wider specimen. This was done to create a larger flow passage under the specimen to reduce the pressure drop which occurred under the narrow specimen.

(4) Installation of a third ion gage. This extra gage permits a more accurate estimation of the pressure directly over the specimen, which pressure is needed for calculation of the "sticking probability" of molecules striking the specimen.

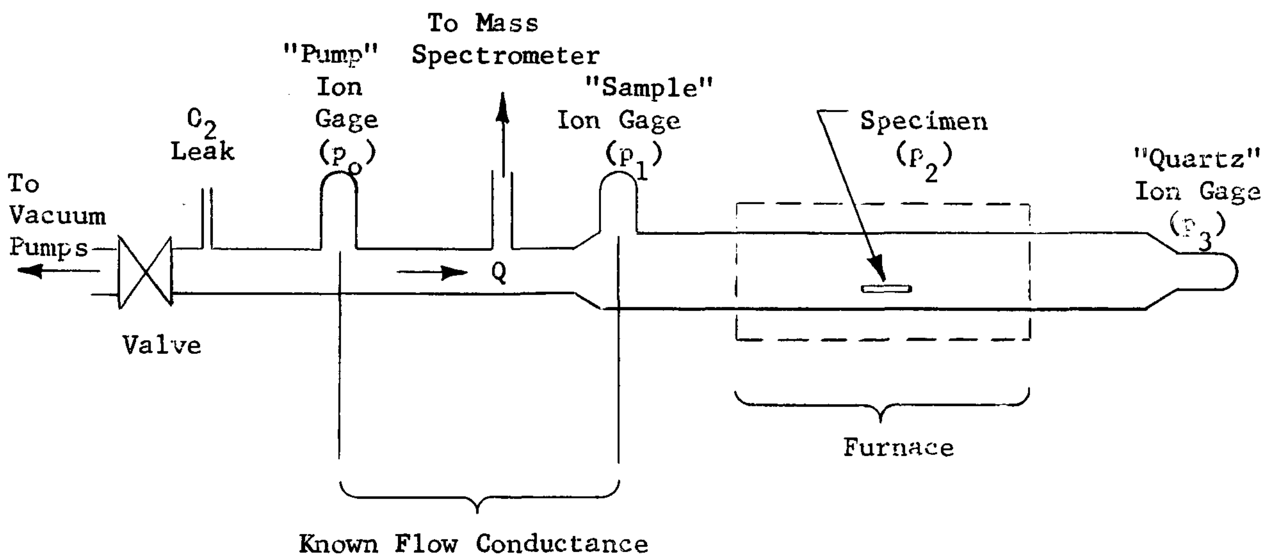
(5) Calibration of ion gages. In the final calibration, two ion gages and their control panels were calibrated by an independent laboratory. These were considered "secondary standards" against which the three ion gages used in the system were calibrated.

The scope of the investigation was confined to exposing the columbium and Cb-1Zr alloy specimens to the various environments and documenting the experimental conditions. Subsequent evaluation of the exposed specimens by chemical and metallurgical techniques will be conducted at the Lewis Research Center of the National Aeronautics and Space Administration. The work performed by the General Electric Company was done at the Advanced Technology Laboratory in Schenectady at the request of the Space Power and Propulsion Section, Re-entry Systems Department, Missile and Space Division.

2. EXPERIMENTAL APPARATUS

The vacuum system used in the recent exposures to oxygen is essentially the same one used under previous NASA contracts. The system is composed of a 115 liter/sec oil diffusion pump, a non-migrating optically tight liquid nitrogen trap and a glass bakeable section comprising a mass spectrometer tube, oxygen permeation leak, ion gage tubes, valve and quartz furnace tube.

Schematically, the system is as follows:



The flow rate is established by measuring the pressure drop (p_o - p_1) across the known conductance. The "quartz" ion gage was installed after the fifth exposure to permit more accurate measurement of the pressure directly over the specimen (p_2).

The ion gage tubes used in the recent exposures were G. E. Model 22GT-102 and contained thoria-coated iridium filaments. The ion gage tubes were operated at 0.1 Ma electron emission. The mass spectrometer tube also contained a thoria-coated iridium filament operating at 1.0 Ma electron emission. The use of the thoria-coated iridium filaments eliminated the task of decarbonizing them, as was done in the past with tungsten, to prevent conversion of O_2 to CO. The use of these new filaments also eliminated the problem of filament loss experienced in other years.

The introduction of oxygen to the system was accomplished with the heated silver permeation membrane, as before. It was found that with constant specimen gettering rate and with approximately one atmosphere of O_2 applied to the leak, the sample ion gage held the desired pressure for many days. The silver membrane leak was found to be relatively insensitive to small O_2 pressure changes on the atmosphere side.

The vacuum system usually reached the low 10^{-8} torr range before O_2 was admitted. This was sufficiently low, inasmuch as the lowest exposure pressure was 10^{-7} torr.

Photographs of the glass system and instrumentation are shown in Figures 1 and 2, on the following pages. The 1100°C furnace is removed in this top view and the recently added ion gage is shown mounted at the end of the quartz tube.

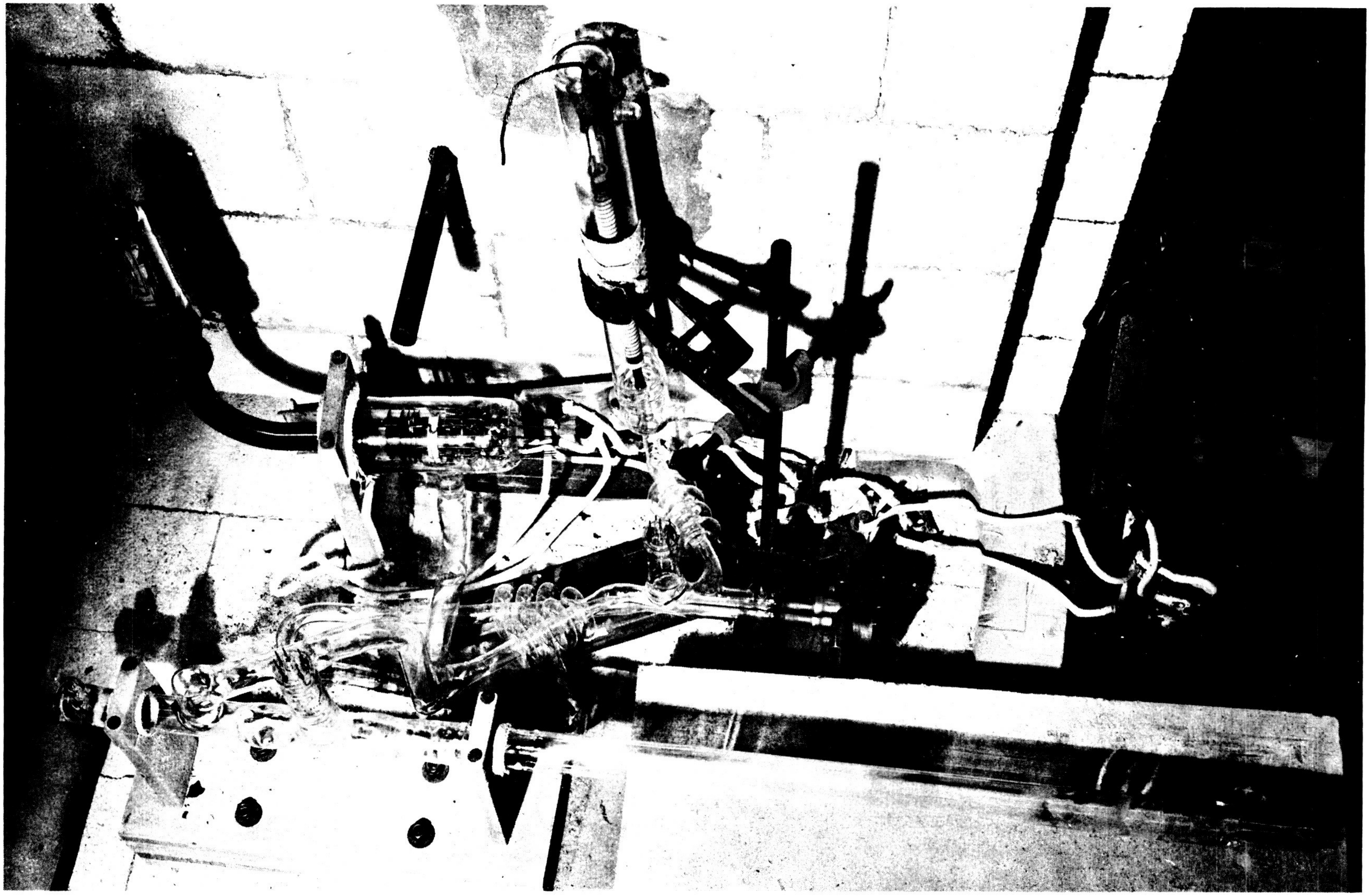


FIGURE 1. PHOTOGRAPH OF GLASS SYSTEM

(ATL Photo 842601)
Mass spectrometer is at upper left, silver O_2 leak at top center. The three ion gages are located as follows: "Pump Gage" (p_o) at center near valve, "Sample Gage" (p_1) at lower left, the recently-installed "Quartz Gage" (p_3) at lower right. The specimen rests in the center of the long quartz tube at the bottom.

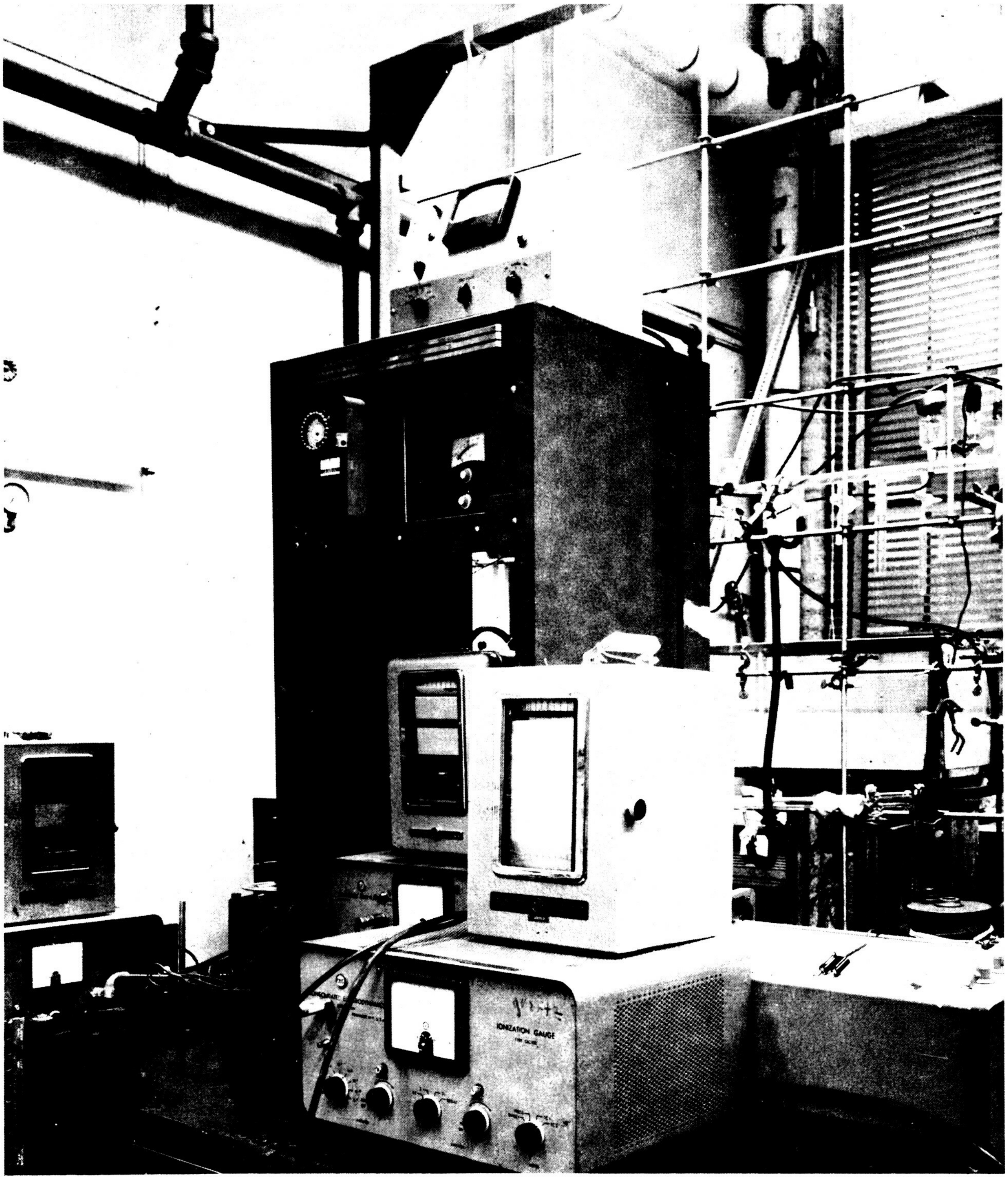


FIGURE 2. ION GAGE CONTROL PANELS

(ATL Photo 842602)

The three Consolidated Vacuum Corp. control panels are shown with the strip-chart recorders, and the x-y plotter for mass spectrometer read-out.

3. GAGE CALIBRATIONS

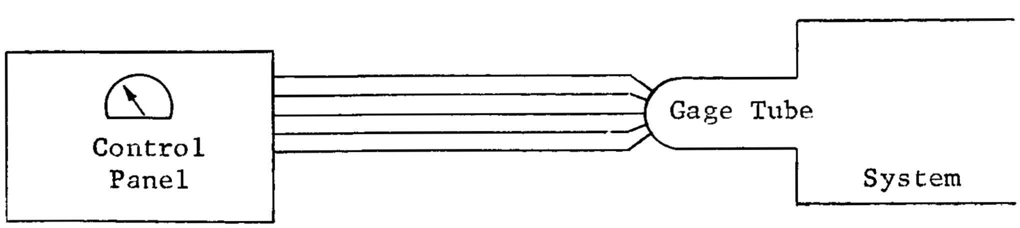
Because the gettering rates established in this program depend directly upon pressure measurements, it is important that the ion gages be carefully calibrated and the readings corrected. The gages were subjected to several calibrations during the year. The earlier calibrations were single-point calibrations of individual gage tubes, and resulted in assigning a "sensitivity" to each of the tubes used in the experiment. This approach assumed identical characteristics and linear scales for each of the gage control panels.

It was later decided to calibrate the gages and panels throughout the range of pressures. This procedure resulted in calibration curves of indicated versus true (corrected) pressures for each gage.

In this Section, there will first be a general discussion of gage calibration, then a description of the earlier calibrations, followed by a discussion of the final calibration. At the end of this Section, there will be a summary of the calibration results for each exposure.

3-1. General Discussion

An ion gage pressure measuring system consists of two elements: an ion gage tube mounted in the system, and a control panel consisting essentially of voltage regulators, a regulated emission current supply and an electrometer circuit for measuring collector current.



The nomenclature to be used in this Section is as follows:

s = Gage sensitivity ($torr^{-1}$)

p = True (corrected) gage pressure (torr)

i_c = True (corrected) collector current (amps)

i_e = True (corrected) emission current (amps)

p_{ind} = Indicated pressure (torr)

i_{c-ind} = Indicated collector current (amps)

i_{e-ind} = Indicated emission current (amps)

F_c = Correction factor for collector current (dimensionless)

F_e = Correction factor for emission current (dimensionless)

The sensitivity of a gage is defined by the following equation:

$$s = \frac{1}{p} \frac{i_c}{i_e} \tag{1}$$

The current correction factors are defined by:

$$i_c = i_{c-ind} F_c \tag{2}$$

and

$$i_e = i_{e-ind} F_e$$

Therefore,

$$s = \frac{1}{p} \frac{i_{c-ind} F_c}{i_{e-ind} F_e} \tag{3}$$

It should be noted that the sensitivity, s, is a characteristic of the gage tube alone, while the correction factors, F_c and F_e are characteristics only of the gage control panels.

The nominal sensitivity of ion gage tubes is 10 $torr^{-1}$. Both the Consolidated Vacuum Corporation panels and the General Electric Company panels which were used in this program are based upon this sensitivity of 10. That is, if $F_c = F_e = 1.0$, and if the gage tube has an actual sensitivity of 10 $torr^{-1}$, then the indicated pressure reading of the panel, p_{ind}, will equal the true (corrected) pressure, p, in torr. For sensitivities other than 10, a correction factor can be obtained from a sensitivity ratio.

As an illustration, if the gage sensitivity for oxygen is 8 per torr, the true (corrected) oxygen pressure will be found from the relationship (assuming again that $F_c = F_e = 1$):

$$p = p_{ind} \frac{10}{8}$$

Thus, if F_c and F_e are unity, the corrected pressure can be found by multiplying the indicated pressure by the ratio of the nominal to the actual gage tube sensitivities.

3-2. Early Calibrations

In the early calibrations, the gages were checked by the Vacuum Products Operation of the General Electric Company, against a "secondary standard" gage, which had previously been calibrated against a McLeod gage. Oxygen was used as the calibrating gas, so a gage sensitivity for oxygen was established directly. The calibration was performed at a single pressure; several references have indicated that gage sensitivities were reasonably constant over the range of pressures used in this program (for example, Davis (1) and Roehrig and Simons (2)).

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(1) W.D. Davis, "Gauge Calibration in the Ultra-high Vacuum Range", Vacuum Symposium Transactions, Amer. Vac. Soc., 1963, p. 253.

(2) J.R. Roehrig and J.C. Simons, "Accurate Calibration of Vacuum Gages to 10^{-9} Torr, "Vacuum Symposium Transactions, Amer. Vacuum Society, 1961, p. 511.

In the calibrations, and in the actual exposures, the correction factors, F_c and F_e were assumed equal to 1.0.

The sensitivities of the gages used for the exposures are summarized in Section 3-4.

3-3. Final Calibration

In order to improve the accuracy of gage calibration, it was decided to calibrate the control panels as well as the gages, thus accounting for deviations of F_c and F_e from 1.0. Two gages were sent to a facility in Boulder, Colorado, for calibration, but because of an apparent inconsistency in the results, the gages were sent to a second facility for recalibration. This final calibration was performed by Varian Associates, Palo Alto, California, in an apparatus described by Hayward and Jepsen (1). Calibrations for both nitrogen and oxygen gas were obtained. Two gages were calibrated with their respective control panels (the gages were General Electric Model 22GT102, and the control panels were General Electric Model 22GC101).

These two gages and control panels were then considered "secondary standards" against which the three gages used in the system were calibrated. The "secondary standards" will not be used in the exposures, but will be saved only for use in gage calibrations. The availability of two secondary standards permits periodic checks of one against the other, increasing the confidence level of any future gage calibrations.

(1) W. H. Hayward and R. L. Jepsen, "A Simple High Vacuum Gauge Calibration System", Vacuum Symposium Transactions, Amer. Vac. Soc., 1962, p. 459.

In addition to the calibration of the two gages with their respective panels by Varian, the General Electric control panels were calibrated independently of the gages, that is, F_c and F_e were established as functions of i_{c-ind}. This makes it possible to compute the sensitivity of the gage alone, using Equation 3, as all factors are now known, since the relationship between the true pressure, p, and the indicated collector current, i_{c-ind}, has been established by Varian. Therefore, either of the secondary standard gages could be used, if necessary, with other control panels of the same voltage characteristics, providing the F_c and F_e of the other panels were known.

The original calibration data produced by Varian are given in the Appendix, Section 5-5.

The method used for calibration of the system gages against the secondary standards is described in the Appendix, Section 5-6.

3-4. Summary of Gage Calibrations

The sensitivities used for each exposure are listed below:

		Gage Sensitivity ($Torr^{-1}$)		
Exposure Number	Specimen Number	Pump Gage (P_o)	Sample Gage (P_1)	Quartz Gage (P_3)
1	34	7.3	6.4	**
2	3	7.3	7.6	**
3	35	7.3	7.3	**
4	1	7.3	7.3	**
5	48	7.3	7.3	**
6	50	7.3	*	*
7	36	*	*	*
8	37	*	*	*
9	52	*	*	*

*Calibration curve given by Figures 20, 21, 22, and 23, in the Appendix, Section 5-6.

**Quartz gage not installed until after 5th exposure.

4. EXPERIMENTAL PROCEDURE AND RESULTS

4-1. Conductance Measurements Between the Pump Ion Gage and Sample Ion Gage

The first experiment was performed to establish experimentally the conductance between the pump ion gage and the sample ion gage. This conductance was established under the previous program, NAS 3-1903,by measuring the glass plumbing between the two ion gages and calculating the conductance using standard formulas.

The current conductance measurement was performed by admitting oxygen to the system through a calibrated leak and recording the difference in pressure between two ion gages. The conductance is based on the relationship $C = \frac{Q}{\Delta p}$ in which

C = Conductance in liters/sec

Q = mass flow in torr liters/sec

p = pressure in torr

The conductance obtained from this experiment was 0.615 liters/sec and compares to 0.604 liters/sec that was calculated under the previous contract. A detailed description of the test procedure is found in the Appendix under 5-1.

Late in the current program of exposures, a more accurate calibration of the ion gage tubes was obtained. With this in mind, it is felt that a more accurate conductance measurement could be realized if the experiment were repeated using this new calibration data.

4-2. Exposures

The same pre-exposure out-gasing procedure was followed in the current nine exposures, that was followed under the two preceding contracts. Before each exposure the glass vacuum system comprising the ion gage tubes, mass spectrometer tube, silver leak, quartz tube and connecting tubes were baked out under vacuum at 400°C for 24 hours. The one non-migrating optically tight liquid nitrogen trap was not baked. At the conclusion of the 400°C bake out, the 980°C to 1100°C furnace was installed around the quartz tube, turned on and approximately two days later with the pressure again in the low 10^{-8} torr range, the specimen was magnetically pushed into the heated quartz tube. After approximately two more days, O_2 was admitted to the low 10^{-8} torr system.

A mass spectrometer gas analysis was made approximately each day of the exposure. Each exposure has a plot or table giving the uncorrected ion gage readings. A gettering rate plot or table is presented in torr liters/sec and is derived from taking the pump ion gage readings (corrected for O_2) and subtracting from it the sample ion gage readings (corrected for O_2). This Δp is multiplied by the conductance between the two gages (0.615 liters/sec). The gas analysis is presented in table form and gives the molar percentage of each gas. The gettering rate plots or tables will be found at the end of this Section 4-2 and the uncorrected pressure plots or tables and the mass spectrometer tables will be found in the Appendix under 5-2.

Exposure No. 1

Narrow Cb-1Zr Specimen No. 34

10^{-6} torr - 980°C - O_2

The mass spectrometer tube was calibrated for CO_2, O_2, CO, N_2 and H_2, before starting the first exposure. A O_2 sensitivity of 7.3 was used for the pump ion gage and 6.4 for the sample ion gage and was derived from the Vacuum Products (G.E.) May calibration. The sample ion gage filaments burned out after the first exposure because of a control panel voltage surge. This hazard was eliminated by installing fuses in the filament leads. The gettering rate data are presented in Figure 3.

Exposure No. 2

Narrow Cb Specimen No. 3

Mid 10^{-5} torr -927°C - O_2

The specimen gettering rate decreased after approximately 100 hours of exposure, and the specimen turned a uniform dull black. Due to the decreased gettering rate, the heat to the silver permeation leak was decreased several times in order to maintain constant pressure.

An O_2 sensitivity of 7.3 was used for the pump ion gage and 7.6 for the sample ion gage. The sample ion gage filament burned out during a calibration and the gage had to be replaced for the third exposure. The gettering rate data are presented in Figure 4.

Exposure No. 3

Narrow Cb-1Zr Specimen No. 35

10^{-5} torr - 1100°C - O_2

A small area of the specimen facing the O_2 flow was oxidized. The pump and sample ion gage tubes were removed after the third exposure and calibrated at Vacuum Products Operation (G. E.). Both gages had O_2 sensitivities of 7.3, and the gettering rate data are presented in Figure 5.

Exposure No. 4

Narrow Cb Specimen No. 1

10^{-6} torr - 980°C - O_2

Both gages had O_2 sensitivities of 7.3, and the gettering rate data are presented in Figure 6.

Exposure No. 5

Wide Cb-1Zr Specimen No. 48

10^{-6} torr - 980°C - O_2

This was the first exposure using a 13/16" wide, 1-3/4" long, 0.0513" thick specimen, and compares to previous specimens measuring 1/2" wide, 3" long and 0.0505" thick. The 0.812" wide specimen sits higher in the 0.825" ID quartz tube, and the flow resistance on opposite sides of the specimen should be more nearly equal than with the 0.5" wide specimen. The 7.3 O_2 sensitivity was again used for both tubes. The gettering rate data are presented in Figure 7.

Exposure No. 6

Wide Cb-1Zr Specimen No. 50

This is the first in a series of four exposures using a third ion gage at the end of the heated quartz tube. The reasons and theory are covered in Section 5-4. This exposure covered pressures from 10^{-5} torr to 10^{-7} torr at 980°C and 1100°C. The true pressure vs panel indicated pressure for the sample and quartz ion gages are given on Figures 20, 21, 22, and 23 of Section

5-6 as derived from the calibration of the gages against the secondary standards. The pump ion gage was replaced because of a leaky graded seal that developed after this 6th exposure.

The 6th exposure "pump" ion gage readings were corrected by using the old 7.3 O_2 sensitivity. The Varian calibration was not received until after the last exposure so the curves of Figures 20, 21, 22, and 23 could not apply to the 6th exposure "pump" ion gage. The mass spectrometer tube was recalibrated after the 6th exposure. The new pump ion gage, sample ion gage, and quartz ion gage were installed in a cluster after the 6th exposure. The system was baked out, O_2 slowly admitted, and the pressure plotted through several decades. The ratios are shown in Figure 19 in the Appendix under 5.6. The gettering rate and pressure data are presented in Table I.

Exposure No. 7

Narrow Cb-1Zr Specimen No. 36

This exposure again covered pressures from 10^{-5} to 10^{-7} torr and 980°C to 1100°C. Two ion gage tubes were sent to Ball Brothers for calibration and the data received were not consistent and have been discarded. The gettering rate and pressure data are presented in Table II. The ion gage pressure readings were corrected to O_2 (torr) by using the plots on Figures 20, 21, 22, and 23 in the Appendix under 5-6.

Exposure No. 8

Narrow Cb-1Zr Specimen No. 37

This exposure covered pressures from 10^{-5} to 10^{-7} torr and 980°C and 1100°C. The gettering rate and pressure data are presented in Table III. The ion gage pressure readings were corrected to O_2 (torr) by using the plots on Figures 20, 21, 22, and 23 in the Appendix under 5-6.

Exposure No. 9

Wide Cb Specimen No. 52

This exposure covered pressures from 10^{-5} torr to 10^{-7} torr and 980°C and 1100°C. Electronic failure in the mass spectrometer power supply prevented analysis of the system in the first phases of the exposure. There was some devitrification of the quartz tube in the area of the specimen because the specimen was approximately 35°C higher than 1100°C in the first three phases of the experiment. The gettering rate and pressure data are presented in Table IV. The ion gage pressure readings were corrected to O_2 (torr) by using the plots on Figures 20, 21, 22, and 23 in the Appendix under 5-6.

FIGURE 3

EXPOSURE OF Cb-1Zr SPEC. NO. 34 AT LOW 10^{-6} TORR AND 980°C WITH O_2 ADMISSION

Exposure No. 1

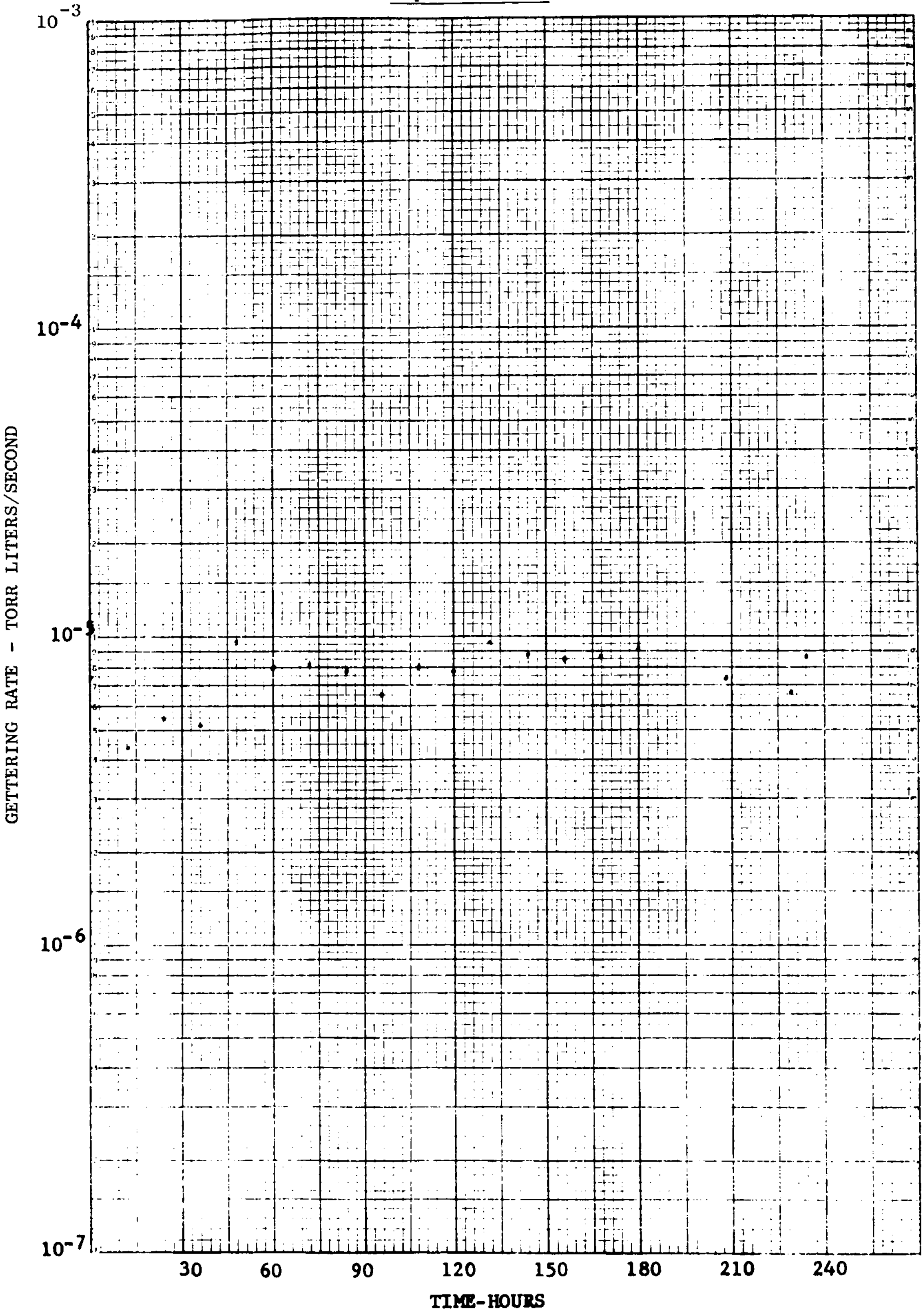


FIGURE 4

EXPOSURE OF Cb SPEC. NO. 3 AT MID 10^{-5} TORR AND 927°C WITH O_2 ADMISSION

Exposure No. 2

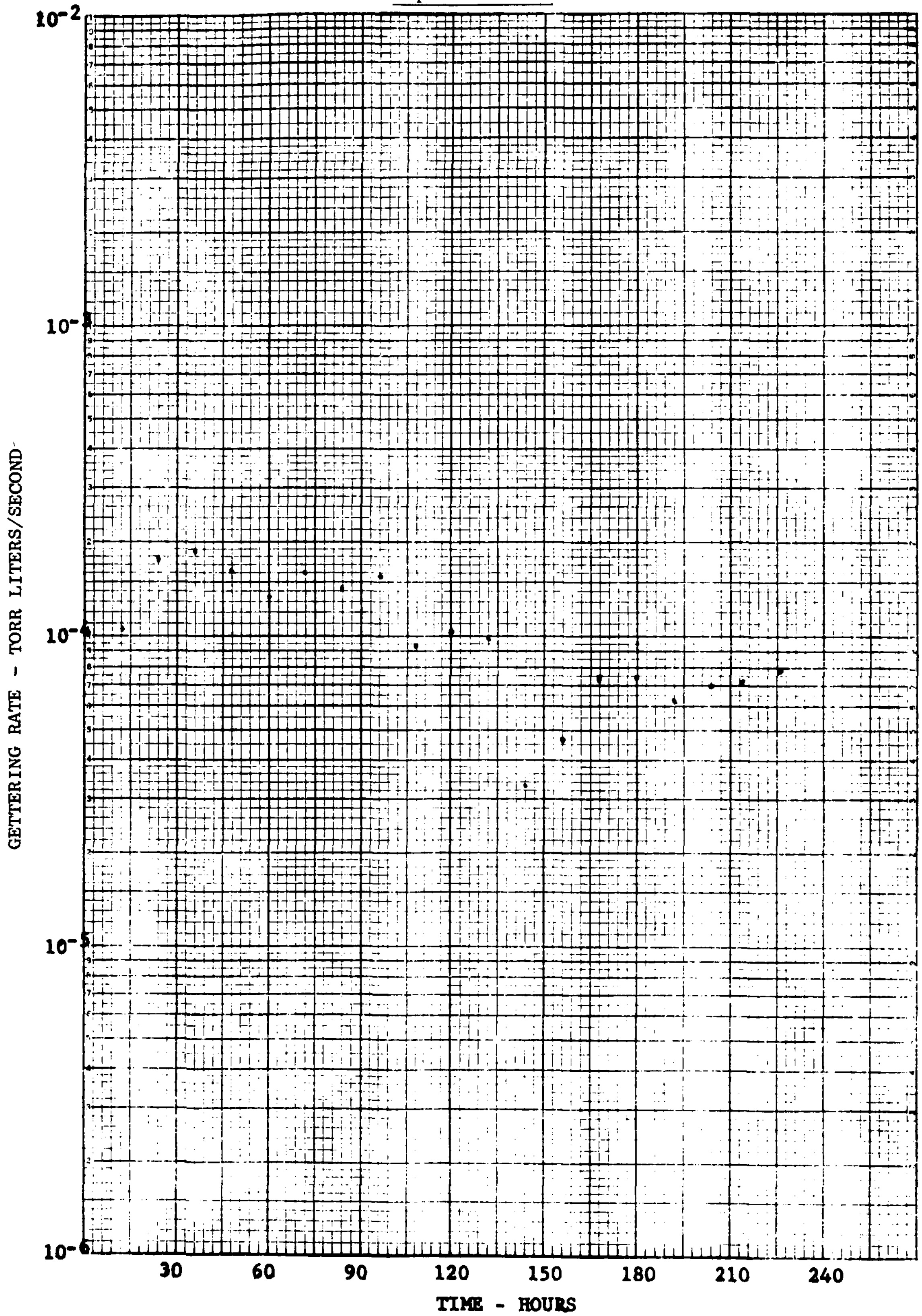


FIGURE 5

EXPOSURE OF Cb-1Zr SPEC. NO. 35 AT LOW 10^{-5} TORR AND 1100°C WITH O_2 ADMISSION

Exposure No. 3

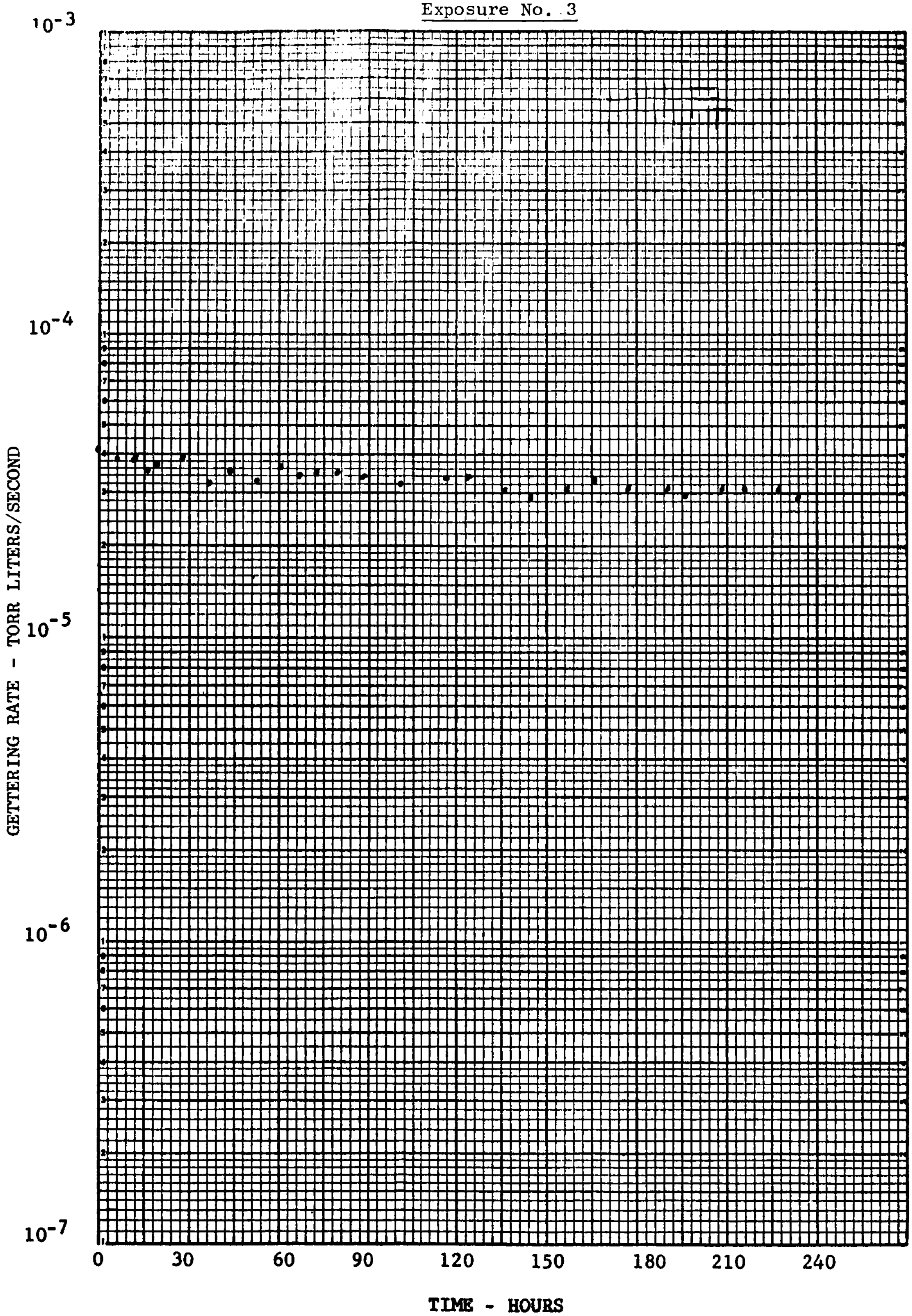


FIGURE 6

EXPOSURE OF Cb SPEC. NO. 1 AT LOW 10^{-6} TORR AND 980°C WITH O_2 ADMISSION

Exposure No. 4

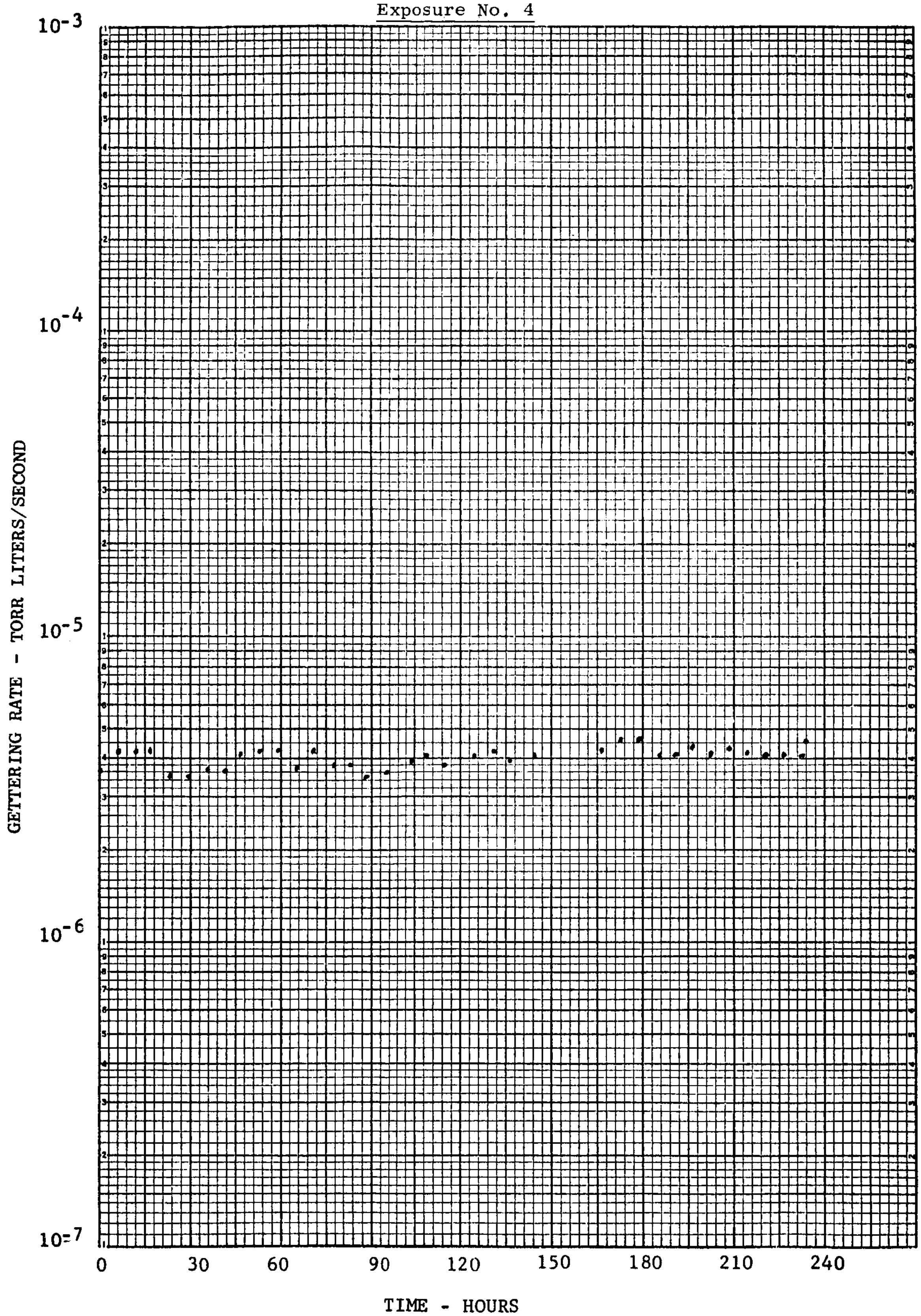


FIGURE 7

EXPOSURE OF Cb-1Zr SPEC. NO. 48 AT LOW 10^{-6} TORR AND 980°C WITH O_2 ADMISSION

Exposure No. 5

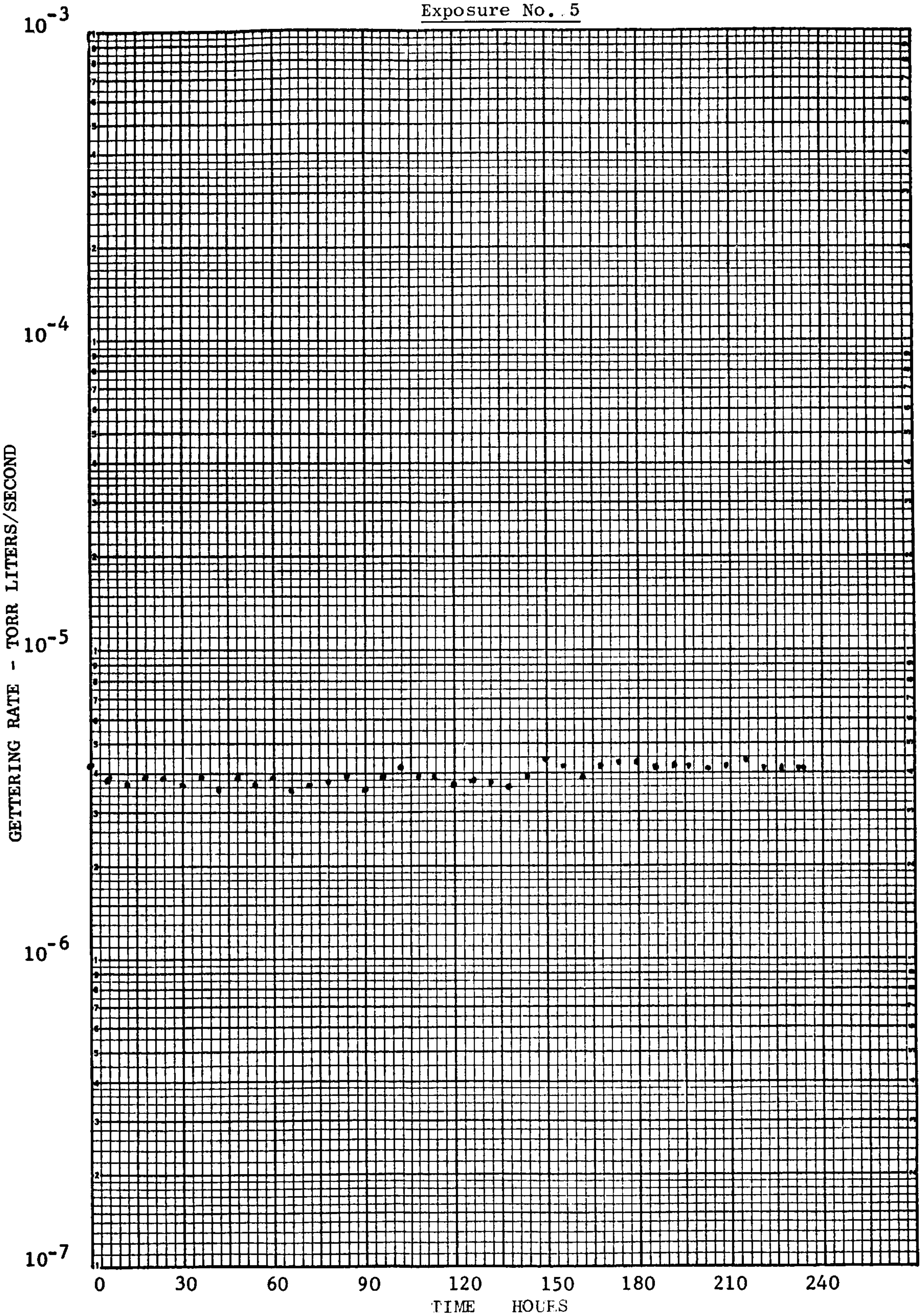


TABLE I

EXPOSURE OF Cb-1Zr SPECIMEN NO. 50

Exposure No. 6

Steady state ion gage readings corrected for 0_2 (torr).

Phase	Specimen Temp.	Pump Ion Gage P_0	Sample Ion Gage P_1	Quartz Ion Gage P_3	$Q=C(P_0-P_1)$ Gettering Rate *	Exposure Time (Hrs)	Notes
1	980°C	8.76×10^{-7}	1.85×10^{-7}	7.3×10^{-8}	4.3×10^{-7}	24	
2	980°C	8.2×10^{-6}	1.36×10^{-6}	2.25×10^{-7}	4.2×10^{-6}	24	
3	1100°C	Low 10^{-8}	Low 10^{-8}	Low 10^{-8}	----	24	0_2 off
4	1100°C	7.25×10^{-7}	1.9×10^{-7}	8.4×10^{-8}	3.29×10^{-7}	72	
5	1100°C	1.18×10^{-5}	1.87×10^{-6}	2.25×10^{-7}	6.1×10^{-6}	24	
6	1100°C	7.8×10^{-5}	1.27×10^{-5}	1.13×10^{-6}	4.01×10^{-5}	24	
7	980°C	Low 10^{-8}	Low 10^{-8}	Low 10^{-8}	---	20	0_2 off
8	980°C	8.22×10^{-5}	1.32×10^{-5}	1.25×10^{-6}	4.25×10^{-5}	24	

* Torr liters/sec.

TABLE II

EXPOSURE OF Cb-1Zr SPECIMEN NO. 36

Exposure No. 7

Steady state ion gage readings corrected for O_2 (torr).

Phase	Specimen Temp.	Pump Ion Gage P_0	Sample Ion Gage P_1	Quartz Ion Gage P_3	$Q=C(P_0-P_1)$ Gettering Rate *	Exposure Time (Hrs)	Notes
1	980°C	$9.6x10^{-7}$	$2.08x10^{-7}$	$5.1x10^{-8}$	$4.63x10^{-7}$	24	
2	980°C	$9.9x10^{-6}$	$1.5\ x10^{-6}$	$1.56x10^{-7}$	$5.17x10^{-6}$	22	
3	980°C	$7.9x10^{-5}$	$1.23x10^{-5}$	$1.08x10^{-6}$	$4.1\ x10^{-5}$	20	
4	1100°C	Low 10^{-8}	Low 10^{-8}	Low 10^{-8}	---	10	O_2 off
5	1100°C	$8.35x10^{-7}$	$1.78x10^{-7}$	$4x10^{-8}$	$4.04x10^{-7}$	18	
6	1100°C	$8.5x10^{-6}$	$1.32x10^{-6}$	$1.2x10^{-7}$	$4.42x10^{-6}$	23	
7	1100°C	$8.0x10^{-5}$	$1.22x10^{-5}$	$1.01x10^{-6}$	$4.17x10^{-5}$	24	
8	1100°C	$4.15x10^{-5}$	$6.7x10^{-6}$	$5.3x10^{-7}$	$2.14x10^{-5}$	24	
9	1100°C	$3.2x10^{-6}$	$5.8x10^{-7}$	$7.9x10^{-8}$	$1.61x10^{-6}$	23	

* Torr liters/sec.

TABLE III

EXPOSURE OF Cb-1Zr SPECIMEN NO. 37

Exposure No. 8

Steady state ion gage readings corrected for O_2 (torr).

Phase	Specimen Temp.	Pump Ion Gage P_0	Sample Ion Gage P_1	Quartz Ion Gage P_3	$Q=C(P_0-P_1)$ Gettering Rate *	Exposure Time (Hrs)	Notes
1	980°C	8.8×10^{-7}	1.84×10^{-7}	3.75×10^{-8}	4.28×10^{-7}	24	(None)
2	980°C	2.22×10^{-6}	4.28×10^{-7}	6.15×10^{-8}	1.1×10^{-6}	24	
3	980°C	4.85×10^{-6}	8.8×10^{-7}	1.2×10^{-7}	2.44×10^{-6}	25	
4	980°C	9.0×10^{-6}	1.4×10^{-6}	1.52×10^{-7}	4.68×10^{-6}	72	
5	980°C	2.58×10^{-5}	4.4×10^{-6}	4.08×10^{-7}	1.32×10^{-5}	23	
6	980°C	4.9×10^{-5}	8.1×10^{-6}	8.3×10^{-7}	2.52×10^{-5}	22	
7	980°C	9.4×10^{-5}	1.43×10^{-5}	1.25×10^{-6}	4.9×10^{-5}	2	

* Torr liters/sec.

TABLE IV

EXPOSURE OF Cb SPECIMEN NO. 52

Exposure No. 9

Steady state ion gage readings corrected for 0_2 (torr).

Phase	Specimen Temp.	Pump Ion Gage P_0	Sample Ion Gage P_1	Quartz Ion Gage P_3	$Q=C(P_0-P_1)$ Gettering Rate *	Exposure Time(Hrs)	Notes
1	1100°C	6.3×10^{-7}	1.73×10^{-7}	7.1×10^{-8}	2.81×10^{-7}	24	(None)
2	1100°C	3.2×10^{-6}	6.1×10^{-7}	1.65×10^{-7}	1.6×10^{-6}	24	
3	1100°C	8.3×10^{-6}	1.36×10^{-6}	3.2×10^{-7}	4.27×10^{-6}	72	
4	1100°C	3.8×10^{-5}	6.0×10^{-6}	1.43×10^{-6}	1.97×10^{-5}	24	
5	1100°C	8.5×10^{-5}	1.94×10^{-5}	2.32×10^{-6}	4.04×10^{-5}	24	
6	980°C	5.4×10^{-7}	1.67×10^{-7}	7.4×10^{-8}	2.3×10^{-7}	24	
7	980°C	2.63×10^{-6}	5.6×10^{-7}	1.75×10^{-7}	1.27×10^{-6}	24	
8	980°C	5.8×10^{-6}	1.74×10^{-6}	3.1×10^{-7}	2.5×10^{-6}	72	
9	980°C	3.7×10^{-5}	6.6×10^{-6}	1.43×10^{-6}	1.87×10^{-5}	24	
10	980°C	9.2×10^{-5}	1.43×10^{-5}	2.8×10^{-6}	4.78×10^{-5}		

* Torr liter/sec.

5. APPENDIX

5-1. Conductance Measurements

In this Section, a detailed description of the conductance measurement, outlined briefly in Section 4-1, will be presented.

The ion gage tubes were installed in the same position as the last contract with the original plumbing between.

To obtain the ΔP between the two ion gages, a nickel calibrated leak was installed as shown on Figure 8. The system was pumped to the mid 10^{-7} torr range with a liquid nitrogen trapped vacuum pump on the atmospheric oxygen side of the leak. This was without the columbium alloy specimen installed and with the 980°C furnace off. The bakeable valve was closed and a small concentration of oxygen was applied to the nickel leak. There was assumed to be no pumping and the two ion gages were plotted through the 10^{-5} torr range as shown in Figure 9.

The bakeable valve was then opened and the pressure decreased to the mid 10^{-7} torr range. Oxygen at one atmosphere was then applied to the nickel leak, and the pump ion gage read 4.9×10^{-5} torr and the sample ion gage read 5.45×10^{-5} torr. Using Figure 9 and making the assumption that the sample ion gage is correct, then when the pump ion gage reads 4.9×10^{-5}, its true corrected reading is 4.2×10^{-5}. The ΔP is therefore 5.45×10^{-5} torr $- 4.2 \times 10^{-5}$ torr $= 1.25 \times 10^{-5}$ torr. If, on the other hand, it is assumed that the pump ion gage is correct, then when the sample ion gage reads 5.45×10^{-5}, its true corrected reading is 6.35×10^{-5} torr. The ΔP is therefore 6.35×10^{-5} torr $- 4.9 \times 10^{-5} = 1.45 \times 10^{-5}$ torr. The average between the two ΔP's is 1.35×10^{-5} torr, and this will be the value used in the conductance calculations.

The conductance is found by the formula:

$$C = \frac{Q}{\Delta P} \qquad \frac{0.83 \times 10^{-5} \text{ torr liters/sec}}{1.35 \times 10^{-5} \text{ torr}}$$

$$C = 0.615 \text{ liters/sec}$$

This compares with the previously calculated conductance of 0.604 liters/sec.

The system used to calibrate the nickel leak for oxygen flow is shown in Figure 10. The equipment consisted of a volume which could be reduced by the introduction of a metal piston through a seal. The piston was pushed into the volume evacuated by the leak. A micrometer head was used to move the piston so that the length of travel could be carefully measured. The piston was introduced into the volume being evacuated at a rate to keep the pressure inside the volume constant. An inclined plane "U" tube manometer, consisting of 10-mil ID glass tubing and using octoil-S as the indicating fluid was used as a pressure gage. This pressure gage was read with a cathetometer and was capable of registering observable pressure changes of approximately 10^{-3} torr at a pressure of one atmosphere.

A precision 0.0623" diameter rod was used and one plot of the penetration rate of this rod is shown. The 0.0623" diameter rod contributes 3.047×10^{-3} cu.in. of volume per inch of penetration. In Figure 11 the penetration is plotted from 0.664" to 0.536" over a 10-minute period, and this contributed 3.9×10^{-4} cu. in. or 6.4×10^{-6} liters in volume. With a difference of one atmosphere of O_2 (or 760 torr) over a ten-minute period, the nickel leak indicates a flow rate of 0.79×10^{-5} torr liter/sec at $20^{o}C$. When the leak was calibrated three different times, the average O_2 flow rate was 0.83×10^{-5} torr liter/sec and this is the value used.

FIGURE 8

FLOW DIAGRAM OF TUBING USED IN CONDUCTANCE CALCULATIONS

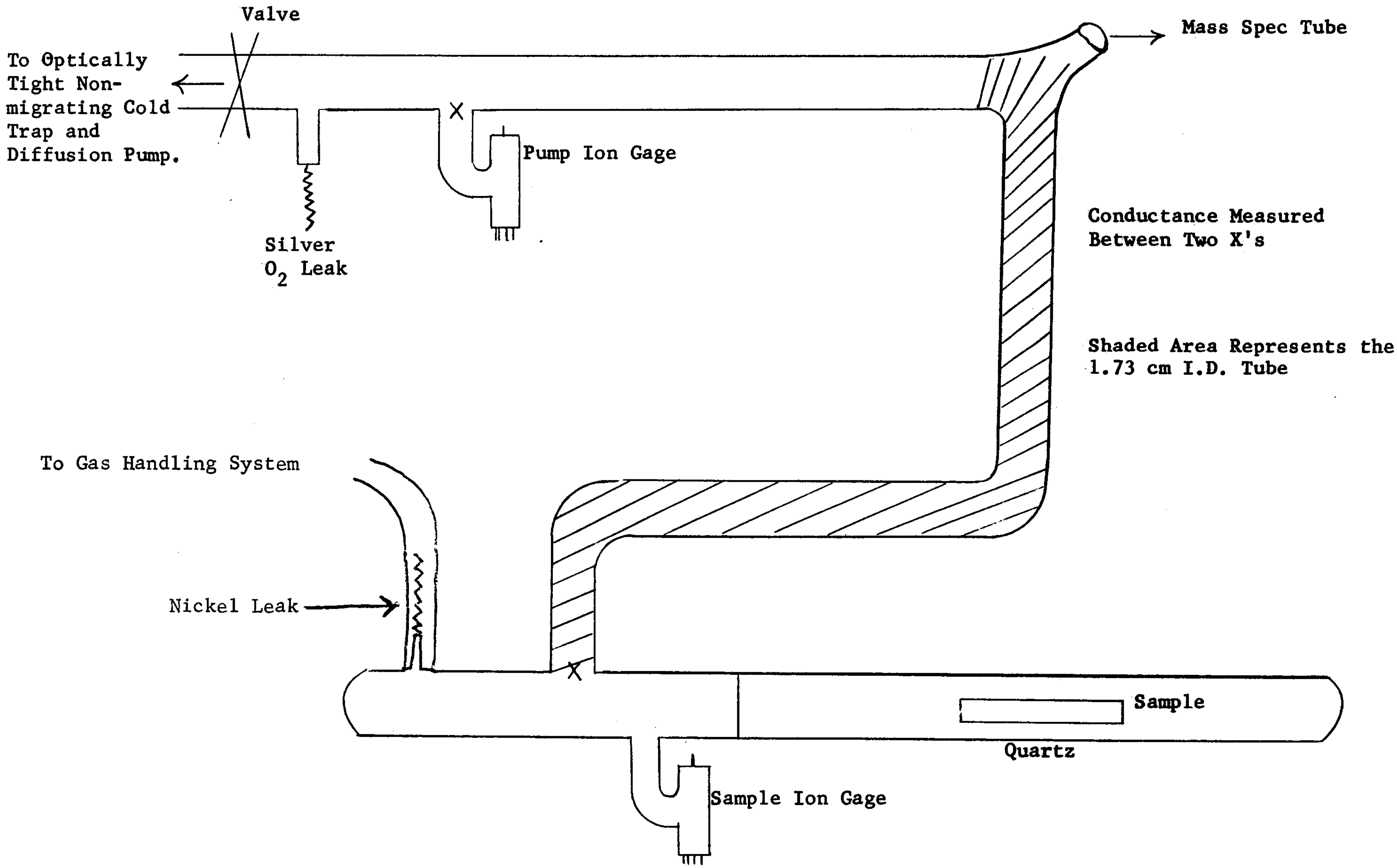


FIGURE 9

ION GAGE COMPARISON - CONDUCTANCE MEASUREMENTS

BAKEABLE VALVE CLOSED

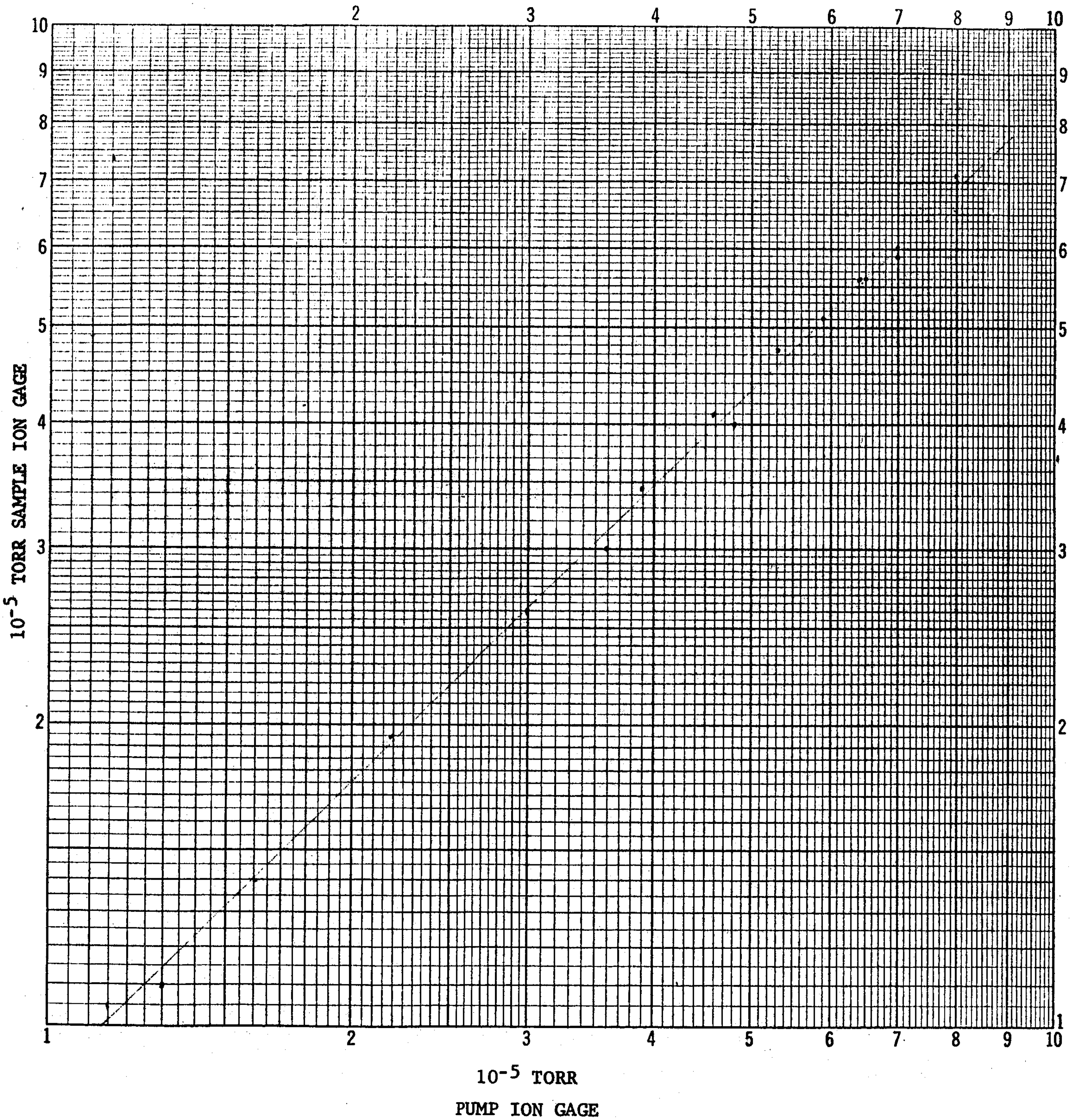
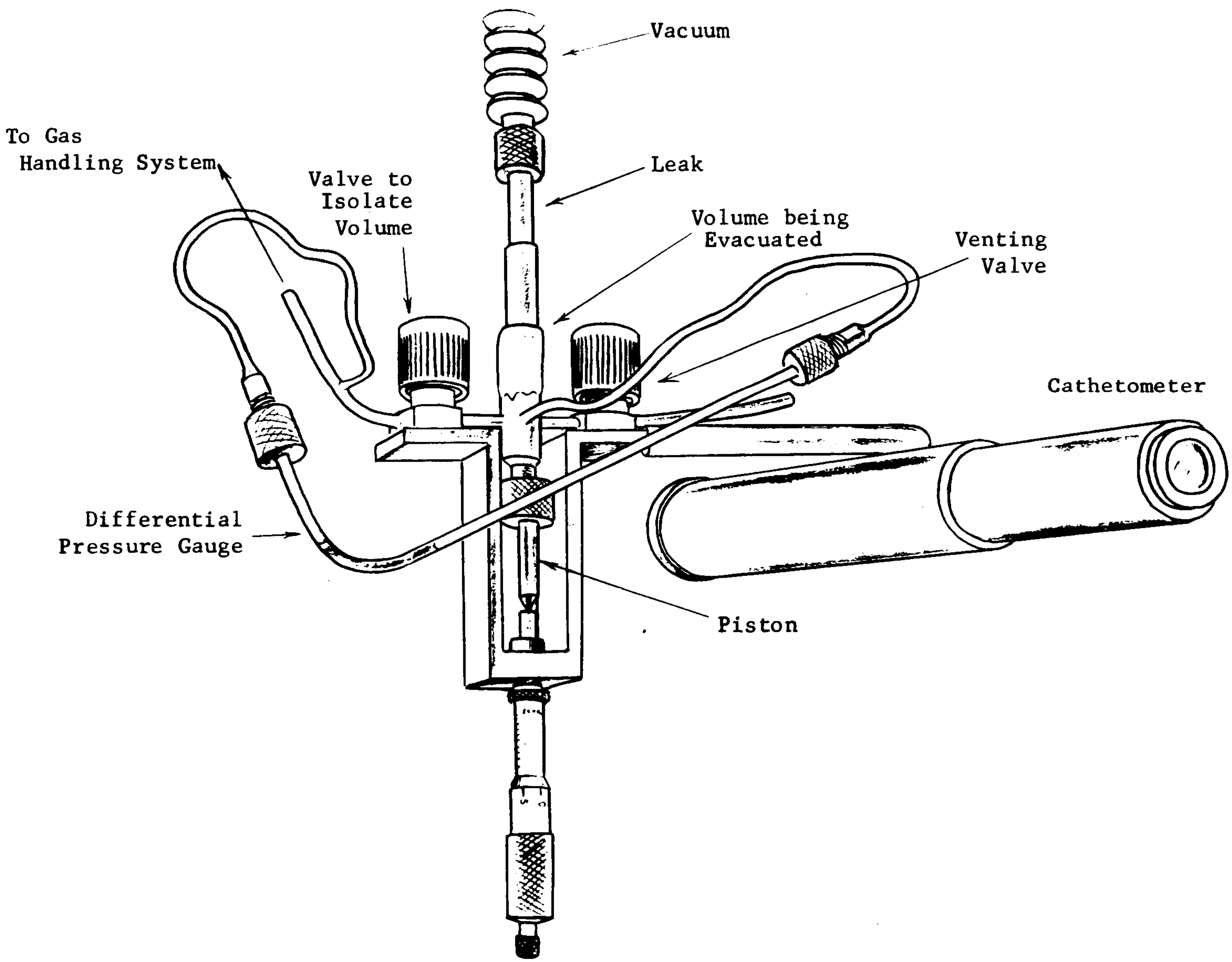


FIGURE 10

LEAK CALIBRATION EQUIPMENT: ISOBARIC VOLUME CHANGE USING MOVABLE PISTON



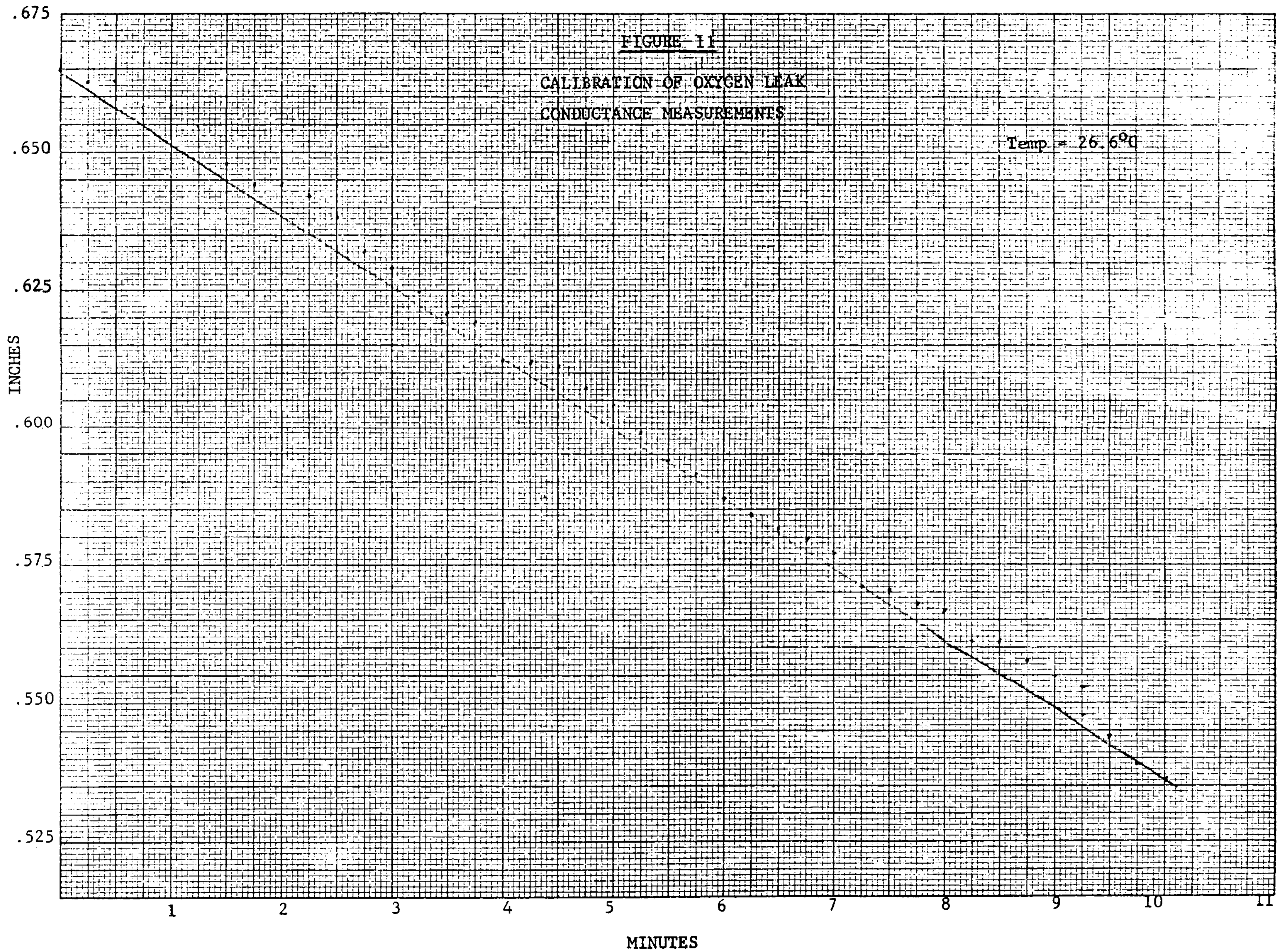


FIGURE 11
CALIBRATION OF OXYGEN LEAK
CONDUCTANCE MEASUREMENTS
Temp = 26.6°C
.675
.650
.625
.600
.575
.550
.525
INCHES
1
2
3
4
5
6
7
8
9
10
11
MINUTES

5-2. Records of Pressure Data and Gas Composition

This Section presents for each exposure, the original uncorrected pressure data and measurements of the gas composition by the mass spectrometer.

The presentation is in two forms. For the first five exposures, when the temperatures and pressures were held constant during each exposure, the pressure data are presented in graphical form. For the remaining exposures, when pressures and temperatures were varied during each exposure, the pressure data are presented in tabular form and represent selected pressures which were obtained after steady-state operation had been attained.

TABLE V

GAS COMPOSITION DURING

THE EXPOSURE OF Cb-1Zr SPECIMEN NO. 34

Low 10^{-6} Torr - 980°C - O_2 Admission

Exposure No. 1

Time, Hrs.*	CO_2	O_2	CO	H_2O	H_2
			Gas Composition, Percent		
22 min.	.44	99.2	.20	.11	.05
16 hrs.	.40	99.4	.07	.09	.04
48 hrs.	.20	99.7	.03	.05	.02
72 hrs.	.16	99.73	.04	.05	.02
91 hrs.	.13	99.76	.04	.05	.02
119 hrs.	.10	99.8	.03	.05	.02
136 hrs.	.10	99.82	.03	.04	.01
160 hrs.	.08	99.84	.03	.04	.01
184 hrs.	.07	99.86	.02	.04	.01
211 hrs.	.04	99.9	.02	.04	-
235 hrs.	.06	99.89	.02	.03	-

* Time from the start of O_2 admission.

FIGURE 12

EXPOSURE OF Cb-1Zr SPEC. NO. 34 AT LOW 10^{-6} TORR AND 980°C WITH O_2 ADMISSION

Exposure No. 1

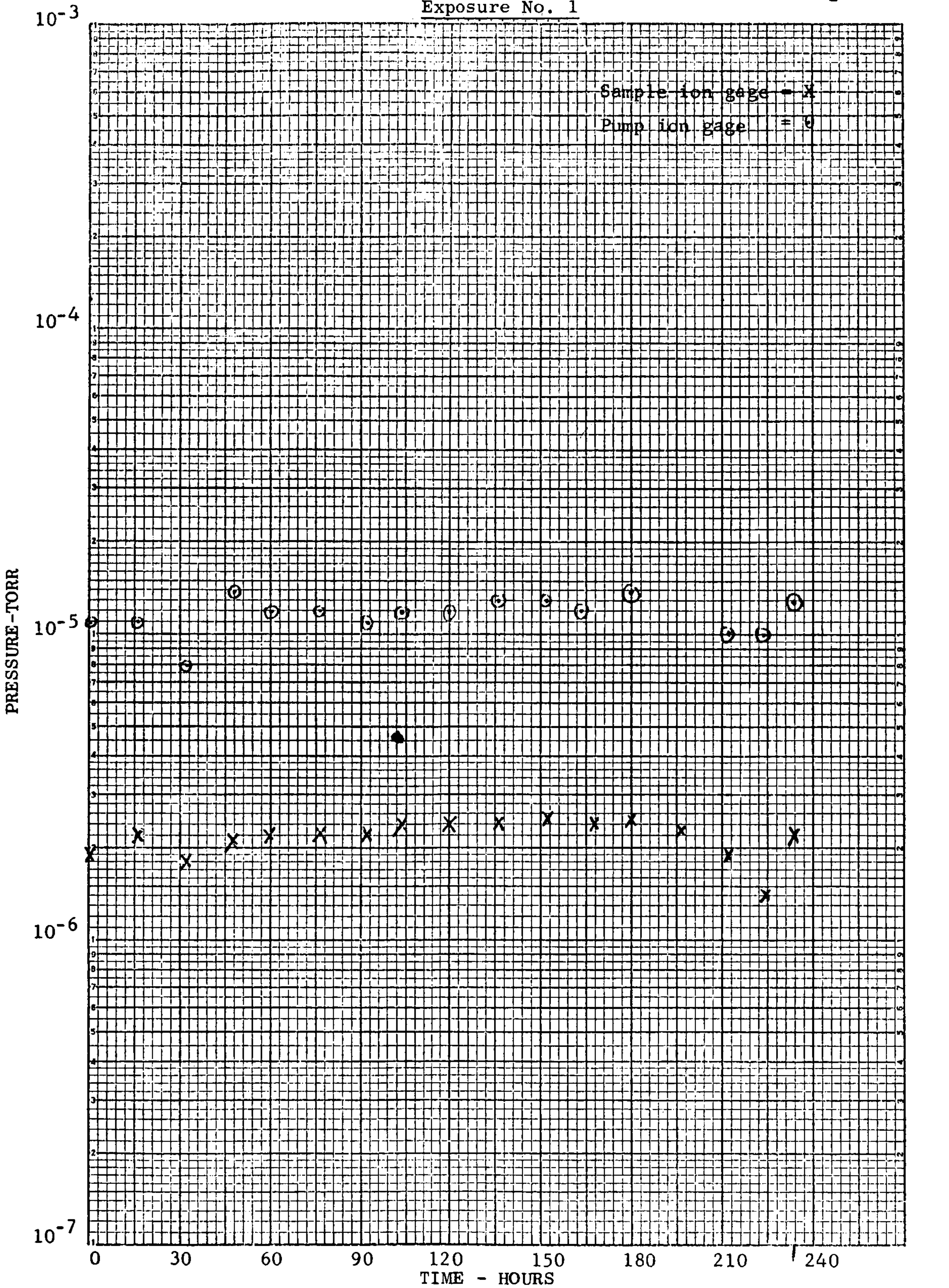


TABLE VI

GAS COMPOSITION DURING

THE EXPOSURE OF Cb SPECIMEN NO. 3

Mid 10^{-5} Torr - 927°C - O_2 Admission

Exposure No. 2

Time, Hrs.*	Gas Composition, Percent			
	CO_2	O_2	CO	H_2O
18	.27	99.7	-	.03
42.5	.25	99.46	.27	.02
72	.27	99.68	.03	.02
87.5	.21	99.75	.02	.02
140	.19	99.77	.02	.02
166.5	.19	99.77	.02	.02
190.5	.16	99.8	.018	.02
216	.16	99.8	.02	.02
233.5	.16	99.8	.02	.02

* Time from the start of O_2 admission.

FIGURE 13

EXPOSURE OF Cb SPEC. NO. 3 AT MID 10^{-5} TORR AND 927°C WITH O_2 ADMISSION

Exposure No. 2

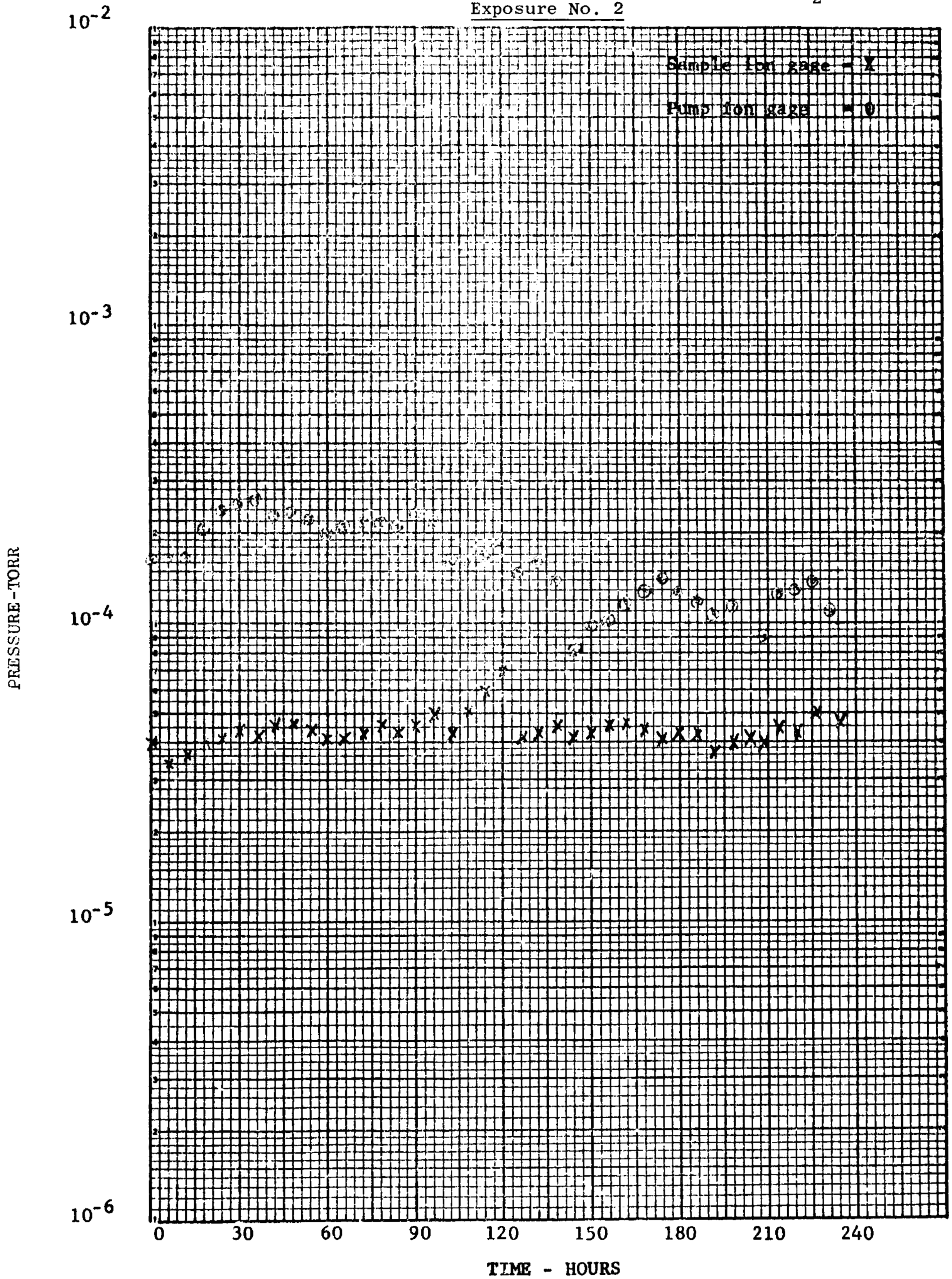


TABLE VII

GAS COMPOSITION DURING

THE EXPOSURE OF Cb-1Zr SPECIMEN NO. 35

Low 10^{-5} Torr - 1100°C - O_2 Admission

Exposure No. 3

Time, Hrs.*	Gas Composition, Percent				
	CO_2	O_2	CO	H_2O	H_2
17.5	.02	99.96	-	.02	-
49	.02	99.96	-	.02	-
73	.02	99.96	-	.02	-
142	.08	99.82	.05	.02	.03
168	.1	99.79	.04	.03	.04
193	.15	99.65	.05	.03	.12
215.5	.2	99.58	.05	.03	.14
235	.24	99.53	.05	.03	.15

* Time from the start of O_2 admission.

FIGURE 14

EXPOSURE OF Cb-1Zr SPEC. NO. 35 AT LOW 10^{-5} TORR AND 1100°C WITH O_2 ADMISSION

Exposure No. 3

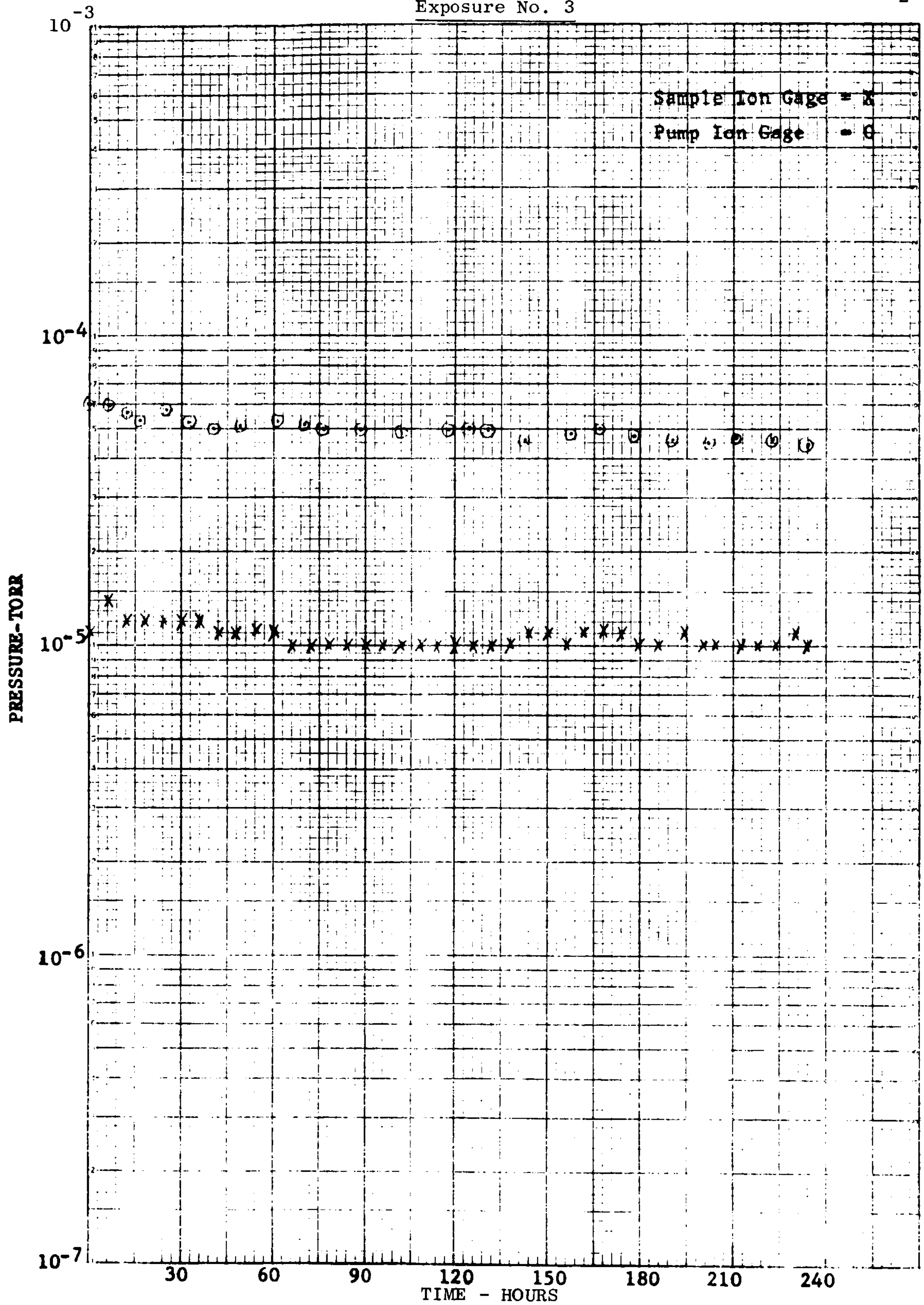


TABLE VIII

GAS COMPOSITION DURING

THE EXPOSURE OF Cb SPECIMEN NO. 1

Low 10^{-6} Torr - 980°C - O_2 Admission

Exposure No. 4

Time, Hrs.*	Gas Composition, Percent				
	CO_2	O_2	CO	H_2O	H_2
23.5	.44	99.31	.07	.10	.08
47.5	.30	99.49	.05	.09	.07
71.5	.2	99.61	.04	.085	.065
138	.12	99.75	.02	.06	.05
169	.096	99.779	.02	.06	.045
193	.098	99.762	.02	.06	.06
233	.073	99.807	.02	.06	.04

* Time from the start of O_2 admission.

FIGURE 15

EXPOSURE OF Cb SPEC. NO. 1 AT LOW 10^{-6} TORR AND 980°C WITH O_2 ADMISSION

Exposure No. 4

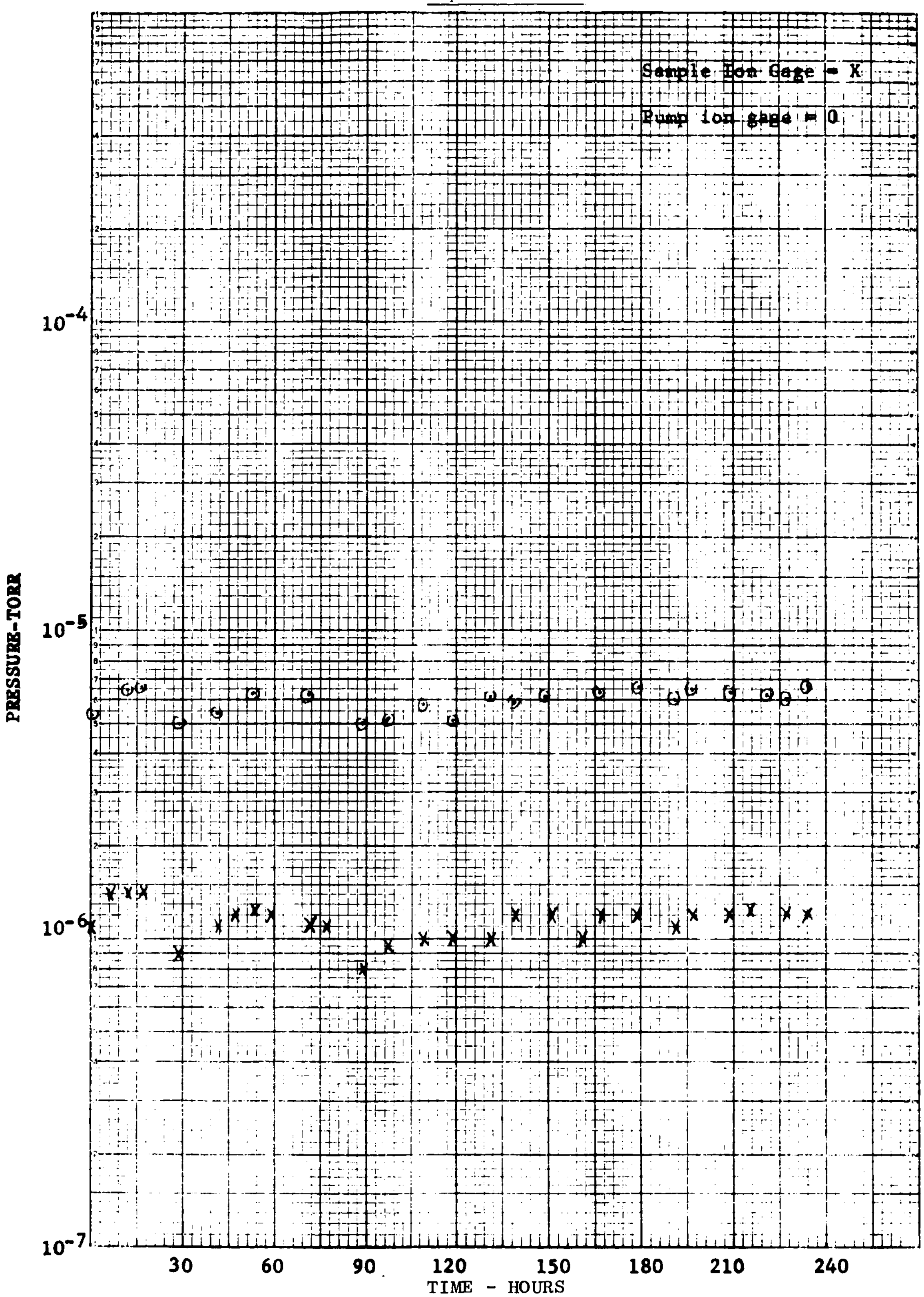


TABLE IX

GAS COMPOSITION DURING

THE EXPOSURE OF Cb-1Zr SPECIMEN NO. 48

Low 10^{-6} Torr - 980°C - O_2 Admission

Exposure No. 5

Time, Hrs.*	Gas Composition, Percent				
	CO_2	O_2	CO	H_2O	H_2
18.5	.3	99.54	.05	.09	.02
46	.16	99.71	.03	.08	.02
70	.12	99.77	.02	.07	.02
96.5	.10	99.79	.02	.07	.02
161	.07	99.84	.02	.05	.02
188	.06	99.87	.01	.05	.01
216	.06	99.87	.01	.05	.01
235.5	.06	99.87	.01	.05	.01

* Time from the start of O_2 admission.

FIGURE 16

EXPOSURE OF Cb-1Zr SPEC. NO. 48 AT LOW 10^{-6} TORR AND 980°C WITH O_2 ADMISSION

Exposure No. 5

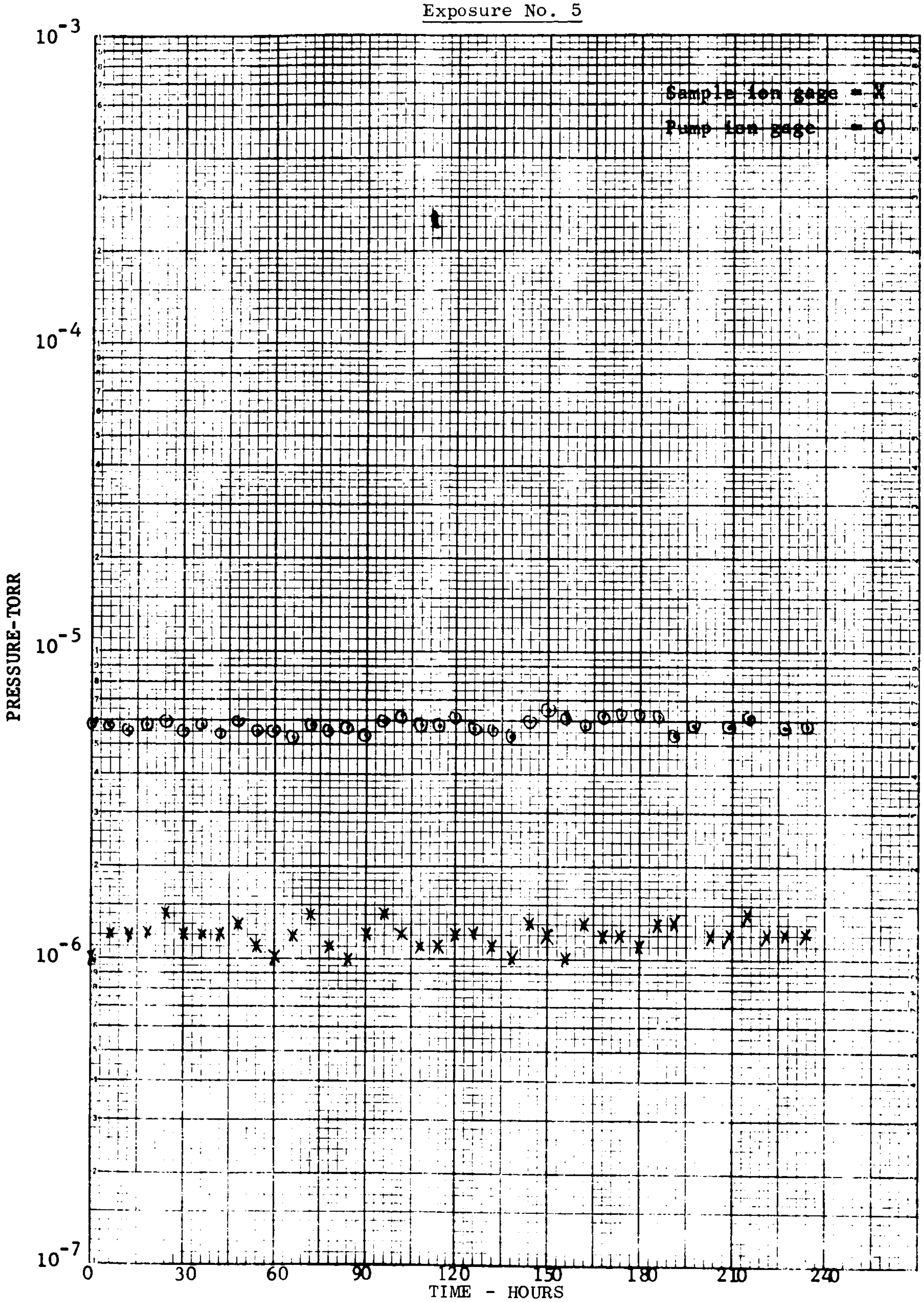


TABLE X

GAS COMPOSITION DURING

THE EXPOSURE OF Cb-1Zr SPECIMEN NO. 50

Exposure No. 6

Exposure	Time, Hrs.*	Gas Composition, Percent					
		CO_2	O_2	CO	H_2O	H_2	H_e
10^{-7} Torr-O_2-980°C	20	4.3	93.87	0.88	.37	0.58	
10^{-6} Torr-O_2-980°C	46	.54	99.3	.07	.08	.01	
10^{-8} -O_2Off-1100°C	66	13.2	11.5	7.20	2.7.	44.0	21.4
10^{-7} -O_2-1100°C	138.5	1.74	96.66	.36	.47	.77	
10^{-6} -O_2-1100°C	166.5	.35	98.88	.62	.1	.05	
10^{-5} -O_2-1100°C	190.5	.1	99.82	.02	.04	.02	
10^{-8} -O_2Off-980°C	211.5	16.3	57.2	6.	3.97	12.3	4.23
10^{-5} -O_2-980°C	235.5	.2	99.76	.02	.02		

* Time from the start of O_2 admission.

TABLE XI

EXPOSURE OF Cb-1Zr SPECIMEN NO. 50

Exposure No. 6

(Readings uncorrected for ion gage sensitivity and gas composition)

8-11-64 3:20 PM O_2 introduced to mid 10^{-8} torr system

Run 10^{-7} torr - 980°C

8-12-64	9:45 AM	Pump ig	6.8×10^{-7} torr
		Sample ig	1.8×10^{-7} torr
		Quartz ig	$.82 \times 10^{-7}$ torr
	1:30 PM	Pump ig	6.4×10^{-7} torr
		Sample ig	1.7×10^{-7} torr
		Quartz ig	$.78 \times 10^{-7}$ torr
	3:06 PM	More O_2 introduced -- Run - 10^{-6} torr - 980°C	
8-13-64	11:05 AM	Pump ig	6.2×10^{-6} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	$.25 \times 10^{-6}$ torr
	1:10 PM	Pump ig	6.0×10^{-6} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	$.24 \times 10^{-6}$ torr
	3:48 PM	O_2 off - Sample up to 1100°C	
8-14-64	8:40 AM	Pump ig	6.2×10^{-8} torr
		Sample ig	5.8×10^{-8} torr
		Quartz ig	2.9×10^{-8} torr
	2:00 PM	Pump ig	5.9×10^{-8} torr
		Sample ig	5.7×10^{-8} torr
		Quartz ig	2.2×10^{-8} torr
	2:18 PM	O_2 in - Run 10^{-7} torr-1100°C	
	5:00 PM	Pump ig	5.4×10^{-7} torr
		Sample ig	1.7×10^{-7} torr
		Quartz ig	$.85 \times 10^{-7}$ torr
8-15-64 Sat.	3:50 PM	Pump ig	5.3×10^{-7} torr
		Sample ig	1.8×10^{-7} torr
		Quartz ig	$.82 \times 10^{-7}$ torr

Table XI (cont'd)

8-16-64 Sun.	1:05 PM	Pump ig	5.4×10^{-7} torr
		Sample ig	1.8×10^{-7} torr
		Quartz ig	$.85 \times 10^{-7}$ torr
8-17-64	1:10 PM	Pump ig	5.3×10^{-7} torr
		Sample ig	1.75×10^{-7} torr
		Quartz ig	$.86 \times 10^{-7}$ torr
	1:32 PM	More O_2 introduced - Run 10^{-6} torr-1100°C	
	4:22 PM	Pump ig	7.7×10^{-6} torr
		Sample ig	1.4×10^{-6} torr
		Quartz ig	$.31 \times 10^{-6}$ torr
8-18-64	8:10 AM	Pump ig	8.2×10^{-6} torr
		Sample ig	1.5×10^{-6} torr
		Quartz ig	$.24 \times 10^{-6}$ torr
	1:33 PM	Pump ig	8.6×10^{-6} torr
		Sample ig	1.6×10^{-6} torr
		Quartz ig	$.24 \times 10^{-6}$ torr
	2:30 PM	More O_2 introduced - Run 10^{-5} torr - 1100°C	
	4:30 PM	Pump ig	6×10^{-5} torr
		Sample ig	1.15×10^{-5} torr
		Quartz ig	$.115 \times 10^{-5}$ torr
8-19-64	8:15 AM	Pump ig	5.9×10^{-5} torr
		Sample ig	1.1×10^{-5} torr
		Quartz ig	$.11 \times 10^{-5}$ torr
	1:30 PM	Pump ig	5.7×10^{-5} torr
		Sample ig	1.05×10^{-5} torr
		Quartz ig	$.11 \times 10^{-5}$ torr
	2:40 PM	O_2 off - Sample 980°C	
8-20-64	8:15 AM	Pump ig	4.9×10^{-8} torr
		Sample ig	4.4×10^{-8} torr
		Quartz ig	6.5×10^{-8} torr
	10:24 AM	Pump ig	4.6×10^{-8} torr
		Sample ig	4.4×10^{-8} torr
		Quartz ig	6.5×10^{-8} torr
	10:59 AM	O_2 in - Run 10^{-5} - 980°C	

Table XI (cont'd)

8-20-64 (cont'd)	2:20 PM	Pump ig	6×10^{-5} torr
		Sample ig	1.1×10^{-5} torr
		Quartz ig	$.13 \times 10^{-5}$ torr
8-21-64	8:10 AM	Pump ig	6×10^{-5} torr
		Sample ig	1.1×10^{-5} torr
		Quartz ig	$.13 \times 10^{-5}$ torr
	11:20 AM	O_2 off - Furnace off	

TABLE XII

GAS COMPOSITION DURING

THE EXPOSURE OF Cb-1Zr SPECIMEN NO. 36

Exposure No. 7

Exposure	Time, Hrs.*	Gas Composition, Percent					
		CO_2	O_2	CO	H_2O	H_2	H_e
10^{-7} Torr-O_2-980°C	23	0.2	97.68	0.36	0.64	0.81	0.31
10^{-6} Torr-O_2-980°C	46.5	0.04	99.70	0.07	0.08	0.05	0.06
10^{-7} Torr-O_2-1100°C	139	0.12	97.38	0.54	0.60	0.93	0.43
10^{-6} Torr-O_2-1100°C	167	0.02	99.68	0.07	0.10	0.04	0.09
10^{-5} Torr-O_2-1100°C	192	0.02	99.92	0.04	0.02	--	--
$5x10^{-6}$ Torr-O_2-1100°C	215	0.02	99.90	0.04	0.04	--	--
$5x10^{-7}$ Torr-O_2-1100°C	234	0.05	99.37	0.18	0.19	0.08	0.13

* Time from the start of O_2 admission.

TABLE XIII

EXPOSURE OF Cb-1Zr SPECIMEN NO. 36

Exposure No. 7

(Readings uncorrected for ion gage sensitivity and gas composition)

9-29-64	3:10 PM	O_2 introduced to mid 10^{-8} torr system	
		Run 10^{-7} - 980°C	
	4:50 PM	Pump ig	6.6×10^{-7} torr
		Sample ig	1.7×10^{-7} torr
		Quartz ig	$.48 \times 10^{-7}$ torr
9-30-64	8:20 AM	Pump ig	6.9×10^{-7} torr
		Sample ig	1.8×10^{-7} torr
		Quartz ig	$.55 \times 10^{-7}$ torr
	3:00 PM	Pump ig	7.4×10^{-7} torr
		Sample ig	1.9×10^{-7} torr
		Quartz ig	$.57 \times 10^{-7}$ torr
	3:30 PM	More O_2 introduced -- Run 10^{-6} torr - 980°C	
	4:23 PM	Pump ig	7.6×10^{-6} torr
		Sample ig	1.3×10^{-6} torr
		Quartz ig	$.175 \times 10^{-6}$ torr
10-1-64	8:00 AM	Pump ig	7.8×10^{-6} torr
		Sample ig	1.4×10^{-6} torr
		Quartz ig	$.165 \times 10^{-6}$ torr
	11:30 AM	Pump ig	7.8×10^{-6} torr
		Sample ig	1.4×10^{-6} torr
		Quartz ig	$.17 \times 10^{-6}$ torr
	1:21 PM	Pump ig	7.5×10^{-6} torr
		Sample ig	1.3×10^{-6} torr
		Quartz ig	$.17 \times 10^{-6}$ torr
	1:50 PM	More O_2 introduced Run 10^{-5} torr - 980°C	
10-2-64	9:35 AM	Pump ig	6.0×10^{-5} torr
		Sample ig	1.0×10^{-5} torr
		Quartz ig	$.11 \times 10^{-5}$ torr
	10:10 AM	Pump ig	5.8×10^{-5} torr
		Sample ig	1.0×10^{-5} torr
		Quartz ig	$.1 \times 10^{-5}$ torr
	10:15 AM	O_2 off - Sample up to 1100°C	

Table XIII (cont'd)

10-3-64 Sat.	7:15 AM	Pump ig	5.1×10^{-8} torr
		Sample ig	3.8×10^{-8} torr
		Quartz ig	2.3×10^{-8} torr
	7:42 AM	O_2 in Run 10^{-7} torr - 1100°C	
10-4-64 Sun.	7:21 AM	Pump ig	8.0×10^{-7} torr
		Sample ig	1.85×10^{-7} torr
		Quartz ig	$.465 \times 10^{-7}$ torr
10-5-64 Mon.	8:00 AM	Pump ig	6.8×10^{-7} torr
		Sample ig	1.7×10^{-7} torr
		Quartz ig	$.46 \times 10^{-7}$ torr
	1:10 PM	Pump ig	6.5×10^{-7} torr
		Sample ig	1.65×10^{-7} torr
		Quartz ig	$.46 \times 10^{-7}$ torr
	1:35 PM	More O_2 introduced Run 10^{-6} torr - 1100°C	
	3:55 PM	Pump ig	7.6×10^{-6} torr
		Sample ig	1.4×10^{-6} torr
		Quartz ig	$.16 \times 10^{-6}$ torr
	4:45 PM	Pump ig	7.5×10^{-6} torr
		Sample ig	1.4×10^{-6} torr
		Quartz ig	$.16 \times 10^{-6}$ torr
10-6-64	8:00 AM	Pump ig	7.0×10^{-6} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	$.135 \times 10^{-6}$ torr
	1:45 PM	Pump ig	6.5×10^{-6} torr
		Sample ig	1.15×10^{-6} torr
		Quartz ig	$.13 \times 10^{-6}$ torr
	2:43 PM	More O_2 introduced Run 10^{-5} torr - 1100°C	
	4:20 PM	Pump ig	6.4×10^{-5} torr
		Sample ig	1.1×10^{-5} torr
		Quartz ig	$.088 \times 10^{-5}$ torr
10-7-64	9:00 AM	Pump ig	6.1×10^{-5} torr
		Sample ig	1.05×10^{-5} torr
		Quartz ig	$.096 \times 10^{-5}$ torr
		Continue 10^{-5} 1100°C	

Table XIII (cont'd)

10-7-64	2:53 PM	Pump ig	5.9×10^{-5} torr
		Sample ig	1.0×10^{-5} torr
		Quartz ig	9.5×10^{-7} torr
	3:13 PM	Cut O_2 back - Run 5×10^{-6} torr - 1100°C	
	4:05 PM	Pump ig	3.2×10^{-5} torr
		Sample ig	5.0×10^{-6} torr
		Quartz ig	5.2×10^{-7} torr
10-8-64	8:35 AM	Pump ig	3.3×10^{-5} torr
		Sample ig	5.2×10^{-6} torr
		Quartz ig	5.5×10^{-7} torr
	2:28 PM	Pump ig	3.1×10^{-5} torr
		Sample ig	5.0×10^{-6} torr
		Quartz ig	5.25×10^{-7} torr
	2:50 PM	Cut O_2 back -Run 5×10^{-7} torr - 1100°C	
10-9-64	8:00 AM	Pump ig	2.75×10^{-6} torr
		Sample ig	5.1×10^{-7} torr
		Quartz ig	8.2×10^{-8} torr
	1:20 PM	Pump ig	2.6×10^{-6} torr
		Sample ig	4.8×10^{-7} torr
		Quartz ig	8.3×10^{-8} torr
	1:30 PM	O_2 off - Furnace off	

TABLE XIV

GAS COMPOSITION DURING
THE EXPOSURE OF Cb-1Zr SPECIMEN NO. 37

Exposure No. 8

Exposure	Time, Hrs.*	Gas Composition, Percent					
		CO_2	O_2	CO	H_2O	H_2	He
1.4×10^{-7} Torr-O_2-980°C	1	1.3	91.50	1.0	0.8	4.5	1.0
3.4×10^{-7} Torr-O_2-980°C	48	0.4	97.57	0.24	1.53	0.1	0.16
7.0×10^{-7} Torr-O_2-980°C	72.5	0.2	99.35	0.15	0.13	0.07	0.10
1.0×10^{-6} Torr-O_2-980°C	144.5	0.1	99.68	0.08	0.08	0.02	0.04
3.0×10^{-6} Torr-O_2-980°C	168	0.08	99.79	0.06	0.06	--	0.01
6.1×10^{-6} Torr-O_2-980°C	193.5	0.07	99.82	0.06	0.04	--	0.01
1.0×10^{-5} Torr-O_2-980°C	217.5	0.01	99.89	0.06	0.04	--	--

* Time from the start of O_2 admission.

TABLE XV

EXPOSURE OF Cb-1Zr SPECIMEN NO. 37

Exposure No. 8

(Readings uncorrected for ion gage sensitivity and gas composition)

10-20-64	1:25 PM	O_2 introduced to mid 10^{-8} system Run 1×10^{-7} torr - 980°C	
10-21-64	8:55 AM	Pump ig Sample ig Quartz ig	7×10^{-7} torr 1.7×10^{-7} torr 4.0×10^{-8} torr
	1:45 PM	Pump ig Sample ig Quartz ig	6.8×10^{-7} torr 1.7×10^{-7} torr 4.4×10^{-8} torr
	1:55 PM	More O_2 introduced - Run 3×10^{-7} torr - 980°C	
	4:55 PM	Pump ig Sample ig Quartz ig	1.8×10^{-6} torr 3.5×10^{-7} torr 6.9×10^{-8} torr
10-22-64	8:55 AM	Pump ig Sample ig Quartz ig	1.9×10^{-6} torr 3.4×10^{-7} torr 6.4×10^{-8} torr
	1:35 PM	Pump ig Sample ig Quartz ig	1.9×10^{-6} torr 3.6×10^{-7} torr 6.7×10^{-8} torr
	2:30 PM	More O_2 introduced - Run 7×10^{-7} torr - 980°C	
	4:15 PM	Pump ig Sample ig Quartz ig	3.9×10^{-6} torr 7.3×10^{-7} torr 1.1×10^{-7} torr
10-23-64	9:05 AM	Pump ig Sample ig Quartz ig	3.9×10^{-6} torr 6.8×10^{-7} torr 1.2×10^{-7} torr
	2:55 PM	Pump ig Sample ig Quartz ig	3.8×10^{-6} torr 7×10^{-7} torr 1.3×10^{-7} torr
	3:20 PM	More O_2 introduced - Run 1×10^{-6} torr - 980°C	

Table XV (cont'd)

10-24-64 Saturday	3:25 PM	Pump ig	6×10^{-6} torr
		Sample ig	1.0×10^{-6} torr
		Quartz ig	1.7×10^{-7} torr
10-25-64 Sunday	12:30 PM	Pump ig	6×10^{-6} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	1.6×10^{-7} torr

10-26-64	3:20 PM	Pump ig	6.8×10^{-6} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	1.65×10^{-7} torr
	3:30 PM	More O_2 introduced - Run 3×10^{-6} torr - 980°C	

10-27-64	9:00 AM	Pump ig	2.1×10^{-5} torr
		Sample ig	3.4×10^{-6} torr
		Quartz ig	4.2×10^{-7} torr
	1:15 PM	Pump ig	2×10^{-5} torr
		Sample ig	3.35×10^{-6} torr
		Quartz ig	4.1×10^{-7} torr
	2:01 PM	More O_2 introduced - Run 7×10^{-6} torr - 980°C	

10-28-64	8:08 AM	Pump ig	3.7×10^{-5} torr
		Sample ig	6×10^{-6} torr
		Quartz ig	7.9×10^{-7} torr
	1:05 PM	Pump ig	3.7×10^{-5} torr
		Sample ig	6.1×10^{-6} torr
		Quartz ig	7.9×10^{-7} torr
	3:45 PM	More O_2 introduced - Run 1×10^{-5} torr - 980°C	

10-29-64	1:00 PM	Pump ig	6.4×10^{-5} torr
		Sample ig	1.15×10^{-5} torr
		Quartz ig	1.65×10^{-6} torr
	2:40 PM	Pump ig	6.5×10^{-5} torr
		Sample ig	1×10^{-5} torr
		Quartz ig	1.45×10^{-6} torr

At this point there was experienced a high voltage breakdown in the mass spectrometer tube. The ion gage readings also changed at this point and it was decided to turn off the O_2 and return to the 10^{-8} torr range. On Nov. 2 at 10:15 AM,

Table XV (cont'd)

0_2 was again introduced and the 1 x 10^{-5} torr run rechecked with the following results:

11-2-64	10:15 AM	Pump ig	7 x 10^{-5} torr
		Sample ig	1.2 x 10^{-5} torr
		Quartz ig	1.3 x 10^{-6} torr
	10:20 AM	More 0_2 introduced - Run 3 x 10^{-5} torr - 980°C	
	1:15 AM	Pump ig	1.6 x 10^{-4} torr
		Sample ig	2.6 x 10^{-5} torr
		Quartz ig	3.5 x 10^{-6} torr
	3:00 PM	Pump ig	1.6 x 10^{-4} torr
		Sample ig	2.6 x 10^{-5} torr
		Quartz ig	3.5 x 10^{-6} torr
	4:45 PM	0_2 Off - Furnace Off	

TABLE XVI

GAS COMPOSITION DURING

THE EXPOSURE OF Cb SPECIMEN NO. 52

Exposure No. 9

Exposure	Time, Hrs.*	CO_2	O_2	CO	H_2O	He	H_2
		Gas Composition, Percent					
1×10^{-7} Torr - O_2-1100°C	24	10.0	82.2	3.0	3.0	1.0	0.8
5×10^{-7} Torr - O_2-1100°C	47	3.3	94.41	0.75	1.3	0.16	0.08
4.5×10^{-7} Torr - O_2-980°C	213	3.0	95.58	0.9	0.5		0.02
1×10^{-6} Torr - O_2-980°C	285	1.0	98.36	0.4	0.15	0.08	0.01
4.9×10^{-6} Torr - O_2 980°C	309	0.7	99.02	0.2	0.08		
1.1×10^{-5} Torr - O_2-980°C	334	1.3	97.71	0.51	0.08	0.4	

* Time from the start of O_2 admission.

TABLE XVII

EXPOSURE OF Cb SPECIMEN NO. 52

Exposure No. 9

(Readings uncorrected for ion gage sensitivity and gas composition)

12-9-64	4:03 PM	0_2 introduced to mid 10^{-8} torr system Run - 1 x 10^{-7} torr 0_2 = 1100°C	
12-10-64	12:00 PM	Pump ion gage	5.1 x 10^{-7} torr
		Sample ion gage	1.6 x 10^{-7} torr
		Quartz ion gage	7.2 x 10^{-8} torr
	1:40 PM	Pump ion gage	5.0 x 10^{-7} torr
		Sample ion gage	1.6 x 10^{-7} torr
		Quartz ion gage	7.6 x 10^{-8} torr
	4:12 PM	More 0_2 introduced - Run - 5 x 10^{-7} torr 0_2 - 1100°C	
12-11-64	9:43 AM	Pump ion gage	2.5 x 10^{-6} torr
		Sample ion gage	5.0 x 10^{-7} torr
		Quartz ion gage	1.9 x 10^{-7} torr
	1:30 PM	Pump ion gage	2.6 x 10^{-6} torr
		Sample ion gage	5.0 x 10^{-7} torr
		Quartz ion gage	1.8 x 10^{-7} torr
	3:58 PM	More 0_2 introduced - Run - 1 x 10^{-6} torr 0_2 - 1100°C	
12-12-64 Sat.			
12-13-64 Sun.	8:55 PM	Pump ion gage	6.3 x 10^{-6} torr
		Sample ion gage	1.3 x 10^{-6} torr
		Quartz ion gage	3.5 x 10^{-7} torr
12-14-64	11:30 AM	Pump ion gage	6.2 x 10^{-6} torr
		Sample ion gage	1.2 x 10^{-6} torr
		Quartz ion gage	3.3 x 10^{-7} torr
	3:27 PM	Pump ion gage	6.3 x 10^{-6} torr
		Sample ion gage	1.2 x 10^{-6} torr
		Quartz ion gage	3.3 x 10^{-7} torr
	4:17 PM	More 0_2 introduced Run 5 x 10^{-6} torr 0_2 - 1100°C	

Table XVII (cont'd)

12-15-64	8:37 AM	Pump ion gage	3.0×10^{-5} torr
		Sample ion gage	4.6×10^{-6} torr
		Quartz ion gage	1.0×10^{-6} torr
	1:45 PM	Pump ion gage	2.85×10^{-5} torr
		Sample ion gage	4.6×10^{-6} torr
		Quartz ion gage	1.0×10^{-6} torr
	4:13 PM	More O_2 introduced - Rum 1×10^{-5} torr O_2 - 1100°C	
12-16-64	8:30 AM	Pump ion gage	6.1×10^{-5} torr
		Sample ion gage	1.1×10^{-5} torr
		Quartz ion gage	2.3×10^{-6} torr
	3:20 PM	Pump ion gage	6.3×10^{-5} torr
		Sample ion gage	1.1×10^{-5} torr
		Quartz ion gage	2.3×10^{-6} torr
	4:10 PM	Cut O_2 back - Run 1×10^{-7} torr - 980°C	
12-17-64	2:00 PM	Pump ion gage	4.7×10^{-7} torr
		Sample ion gage	1.6×10^{-7} torr
		Quartz ion gage	8.1×10^{-8} torr
	4:07 PM	Pump ion gage	4.3×10^{-7} torr
		Sample ion gage	1.55×10^{-7} torr
		Quartz ion gage	7.9×10^{-8} torr
	4:15 PM	More O_2 introduced - Run 5×10^{-7} torr O_2 - 980°C	
12-18-64	10:25 AM	Pump ion gage	2.1×10^{-6} torr
		Sample ion gage	4.55×10^{-7} torr
		Quartz ion gage	1.9×10^{-7} torr
	2:30 PM	Pump ion gage	2.2×10^{-6} torr
		Sample ion gage	4.6×10^{-7} torr
		Quartz ion gage	1.9×10^{-7} torr
	4:00 PM	More O_2 introduced - Run 1×10^{-6} torr - 980°C	
12-21-64	9:13 AM	Pump ion gage	4.6×10^{-6} torr
		Sample ion gage	1.0×10^{-6} torr
		Quartz ion gage	3.2×10^{-7} torr

Table XVII (cont'd)

12-21-64 (cont'd)	2:00 PM	Pump ion gage	4.5×10^{-6} torr
		Sample ion gage	1.0×10^{-6} torr
		Quartz ion gage	3.2×10^{-7} torr
	3:13 PM	More O_2 introduced - Run 5×10^{-6} torr - 980°C	
12-22-64	1:17 PM	Pump ion gage	2.9×10^{-5} torr
		Sample ion gage	4.9×10^{-6} torr
		Quartz ion gage	1.55×10^{-6} torr
	3:45 PM	Pump ion gage	2.8×10^{-5} torr
		Sample ion gage	4.9×10^{-6} torr
		Quartz ion gage	1.5×10^{-6} torr
	3:45 PM	More O_2 introduced - Run 1×10^{-5} torr - 980°C	
12-23-64	8:35 AM	Pump ion gage	6.3×10^{-5} torr
		Sample ion gage	1.1×10^{-5} torr
		Quartz ion gage	2.6×10^{-6} torr
	1:40 PM	Pump ion gage	6.8×10^{-5} torr
		Sample ion gage	1.2×10^{-5} torr
		Quartz ion gage	2.7×10^{-6} torr
		O_2 off - Quartz furnace off.	

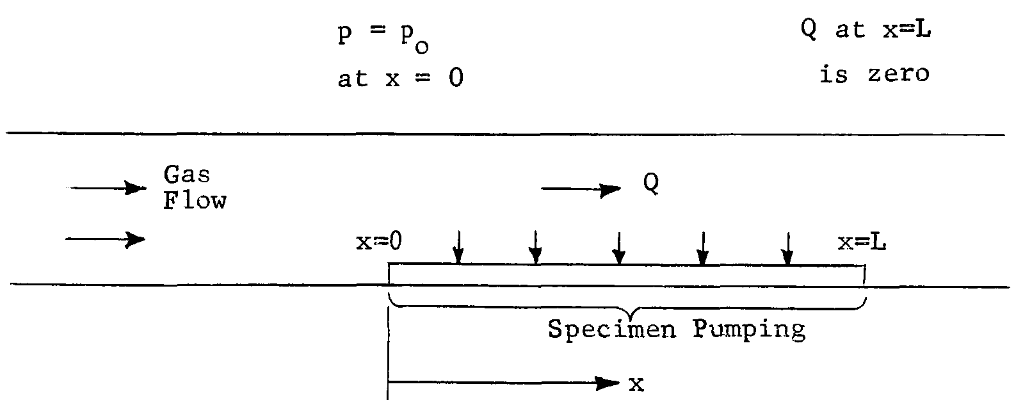
5-3. Pressure Distribution Along Specimen

It was desired to determine if there was a significant pressure change along the length of the specimen. The problem of particular interest was the pressure change along the underneath side of the narrow specimen, because that side of the specimen presented a smaller flow passage, and consequently higher flow resistance and higher pressure drop.

The calculation carried out below indicated that the pressure drops by about 4 to 5% of the initial value along the specimen on the top side. However, the pressure at the center of the narrow specimen on the underneath side may drop to about 38% of the pressure at each end. It was this consideration which led to the use of a wider specimen for the later work in the program.

The calculations on which these conclusions were based are given below.

The nomenclature is given in the following sketch and in the list of symbols following.



Q = the (local) longitudinal mass flow rate (torr liters/sec).

S' = the pumping speed of the specimen per unit length (liters $sec^{-1} cm^{-1}$) (assume a constant)

p = the local pressure (torr)

R = Reciprocal conductance or resistance (sec/liter)

R' = the flow resistance of the duct <u>per unit length</u> (sec liter^{-1} cm^{-1})

$\alpha \equiv \sqrt{R'S'}$ (cm^{-1})

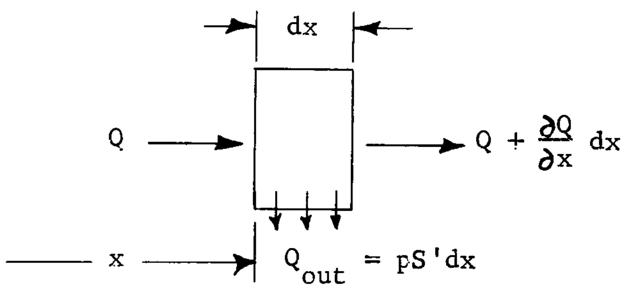
p_o = pressure at $x = 0$

C = Conductance, defined as $C = Q/\Delta p$

T = absolute temperature (^oK)

M = Molecular weight (g/mole)

A differential element of the tube will be drawn:



Equating mass in to mass out gives

$$Q = Q + \frac{\partial Q}{\partial x} dx + pS'dx$$

or,

$$\frac{dQ}{dx} = -pS' \qquad (1)$$

The definition of conductance (or reciprocal resistance)gives

$$Q = C\Delta p = \frac{1}{R}\Delta p$$

Since $\frac{dp}{dx}$ is negative in the direction of flow:

$$Q = -\frac{1}{R'} \frac{dp}{dx}$$

Differentiating,

$$\frac{dQ}{dx} = -\frac{1}{R'} \frac{d^2p}{dx^2} \qquad (2)$$

Combining (2) and (1) gives the basic equation:

$$\frac{d^2p}{dx^2} - R'S'p = 0 \qquad (3)$$

The general solution to this is:

$$p = A\,e^{-\alpha x} + Be^{+\alpha x} \qquad (4)$$

where A & B are constants, and $\alpha \equiv \sqrt{R'S'}$

The boundary conditions are:

(1) at $x = 0$, $p = p_o$ (specified by the conditions of the run)

(2) at $x = L$, $\frac{dp}{dx} = 0$ (no mass flow beyond this point)

The second condition gives $B = A\,e^{-2\alpha L}$ (5)

and the first condition gives

$$A = \frac{p_o}{1 + e^{-2\alpha L}} \qquad (6)$$

Substituting (5) and (6) into (4) gives the final equation:

$$p = \frac{p_o}{1 + e^{-2\alpha L}} \left(e^{-\alpha x} + e^{-2\alpha L}\,e^{\alpha x}\right) \qquad (7)$$

at $x = L$:

$$\frac{p}{p_o} = \frac{2\,e^{-\alpha L}}{1 + e^{-2\alpha L}} \qquad (8)$$

In our particular case the duct is 2.13 cm I.D. and the specimen length is 3 in (= 7.6 cm). The pumping speed is about 3 liters/sec for a typical case. Therefore:

$$S' = \frac{3}{7.6} = 0.4 \ \frac{\text{liters}}{\text{sec cm.}}$$

The conductance of a circular duct is (ignore the effect of the specimen)

$$C = 12.1\ \frac{D^3}{L}\left(\frac{29\ T}{293\ M}\right)^{1/2} \ \frac{\text{liters}}{\text{sec.}}$$

for O_2 at 1000^oC (= 1273 ^oK) this becomes

$$C = 24\ \frac{D^3}{L}$$

$$\therefore\ R = \frac{L}{24\ D^3}, \quad \text{and } R' = \frac{1}{24\ D^3} \qquad \frac{\text{sec}}{\text{liter cm}}$$

In our case then $R' = 4.3 \times 10^{-3}$

then $\alpha \equiv \sqrt{R'S'} = 4.1 \times 10^{-2}\ \frac{1}{\text{cm}}$

from eqn. (8):

$$\frac{p}{p_o} = 0.955 \qquad \text{at } x = L$$

So the pressure drops about 4 or 5% from the beginning to the end of our specimen.

However, the resistance underneath the specimen is greater because of the restricted passage. Data are not available for the conductance of a passage with cross-section boundaries consisting of an arc of a circle and a chord. Therefore the passage will be approximated by a rectangle of dimensions 1.2 cm and 0.2 cm.

For a rectangular duct of large dimension a, and small dimension b,

$$C = 36.7\ \frac{ab^2}{L}\ \ln\ \frac{L}{b} \left(\frac{29}{293}\ \frac{T}{M}\right)^{1/2}$$

Let $T = 1273\ ^oK$

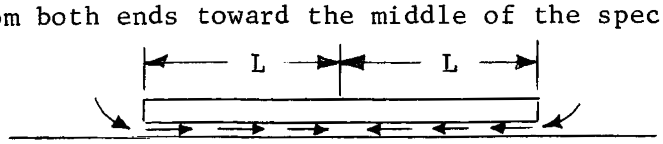
and $M = 32$

$$R = \frac{1}{C} \quad \text{and } R' = \frac{1}{73\ ab^2\ \ln \frac{L}{b}}$$

$$R' = 0.45 \quad \frac{\text{sec}}{\text{liter cm}}$$

Speed = say, 1/2 of 3 or 1.5 liters/sec

Assume pumping from both ends toward the middle of the specimen:



Therefore, $L = \frac{1}{2} \times 7.6 = 3.8$ cm.

and $S' = \frac{1.5}{3.8} = 0.4 \frac{\text{liters}}{\text{sec cm}}$

then $\alpha = \sqrt{R'S'} = 0.425 \frac{1}{\text{cm.}}$

From Eqn. (8):

$$\frac{p}{p_o} = 0.38 \qquad \text{at } x = L$$ (or in this case at the center of the specimen)

Thus, the pressure at the center of the specimen, on the underneath, is calculated to be about 38% of the pressure at the ends.

5-4. Analysis of Pressure Measurements

The installation of the third ion gage (identified as the "quartz" gage) in the system provided an additional means for estimating the pressure directly over the specimen. This third gage was installed prior to the sixth exposure. In the earlier exposures, the specimen pressure was estimated by knowing the pressure of the sample gage, calculating the pressure drop through the conductance between the sample gage and the specimen, and applying a thermal transpiration correction.

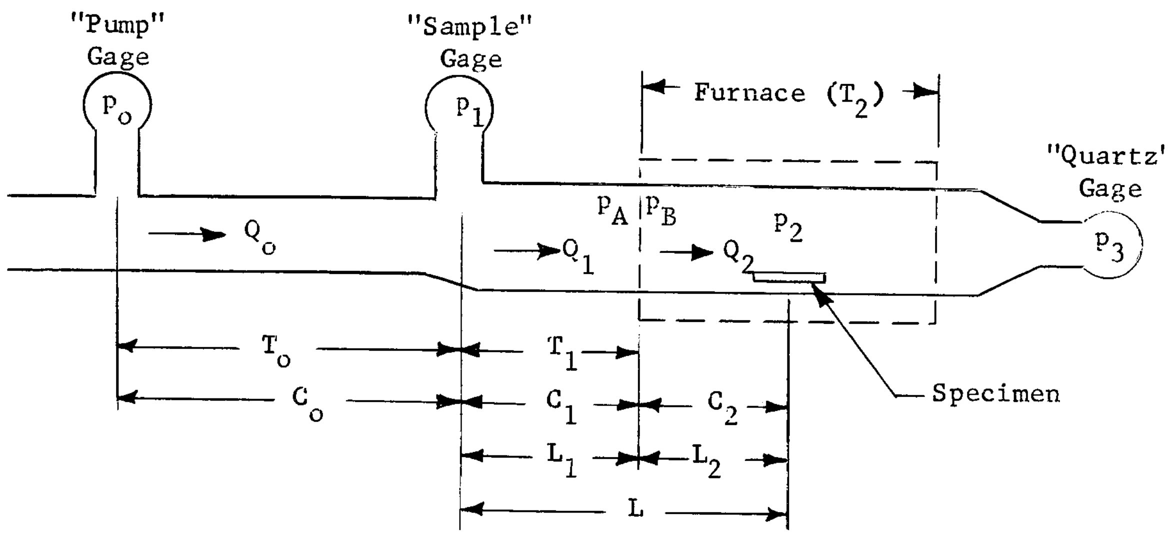
The pressures measured by the quartz gage during the sixth exposure were used to predict the pressure existing directly over the test specimen. The only correction thought to be necessary in converting from the gage measurement to the specimen pressure was that of thermal transpiration, as there should be no net flow of gas between the specimen and gage.

The pressure over the specimen was also predicted from sample gage measurements, using the conventional thermal transpiration and flow-resistance pressure-drop calculations. To our dismay, there was a very large discrepancy between the specimen pressure as predicted by the sample gage and the quartz gage calculations. In fact, pressures predicted from the sample gage measurements were higher than the quartz gage prediction by a factor of up to about 6 at a pressure level of 10^{-5} torr. This pressure ratio of 6 decreased linearly to almost 1.0 as the pressure decreased to 10^{-7} torr (the linear plot was obtained when the ratio was plotted against the log of the pressure level).

It was therefore apparent that a more detailed analysis of the flows and pressures would have to be made. Several methods of analysis were tried, and the one presented below, while not completely satisfactory in every respect, does give fairly consistent results, and should be an aid in correlating the data.

Nomenclature

The sketch below and the following list will define the symbols used.



A = Tube cross-sectional area (cm^2)

C = Conductance, defined by $Q = C\,\Delta p$ (liters/sec)

D = Internal tube diameter (cm)

G = Mass flux ($g\ sec^{-1}\ cm^{-2}$)

K = Constant defined by Eqn. (16)

L = Length, along tube axis (cm)

$\dot{m}$ = Mass flow rate (g/sec)

M = Molecular weight (g/mole)

p = Pressure (torr)

Q = Flow rate in terms of pressure x volume flow rate (proportional to mass flow rate at a given temperature) (torr liters/sec)

R = Gas constant (62.3 torr liter $mole^{-1}$ deg K^{-1}) or, in Eqn.(3), (8.3×10^7 dyne cm $mole^{-1}$ deg K^{-1})

T = Absolute temperature (deg K)

$\dot{V}$ = Volume flow rate (liters/sec)

λ = Pressure multiplier, defined in table

Analysis

The flow rate, Q, is defined by $Q = p\dot{V}$. Since the perfect gas law gives

$$p\dot{V} = \frac{\dot{m}RT}{M} \quad ,$$

the flow rate can be expressed as

$$Q = \frac{\dot{m}RT}{M} \tag{1}$$

Along a tube with axial variations in temperature, the mass flow rate ($\dot{m}$) remains a constant regardless of the temperature. However, the flow rate, Q, varies with temperature. If Q_o = the flow rate where the temperature is T_o, and Q_n = the flow rate where the temperature is T_n,

$$Q_n = \frac{\dot{m}RT_n}{M} = \frac{\dot{m}RT_o}{M} \times \frac{T_n}{T_o}$$

or,

$$Q_n = Q_o \frac{T_n}{T_o} \tag{2}$$

An assumption will now be made about the nature of gas flow in the tube. Because the pressure is low enough, molecular flow will take place in which molecule-molecule collisions will be very rare, and in which the molecules will skip back and forth between the tube walls. Molecules will leave the wall with a cosine distribution, which states that the direction of greatest likelihood is straight across the tube; only a small percentage of molecules will travel straight down the axis of the tube and not hit the wall within a few tube diameters after leaving the wall. With this picture in mind it seems reasonable that if there is a sudden axial change in wall temperature (as at the edge of the furnace), and if the thermal accommodation coefficient is nearly 1.0, the gas traveling down a tube will immediately assume the new wall temperature as it passes a temperature discontinuity.

This will be assumed. To account for those molecules which travel down the tube and hit the specimen without first coming to the wall temperature would make the analysis more complicated than can be justified at this time.

As the gas flowing from left to right passes the edge of the furnace, the gas temperature increases from T_1 to T_2. It would not be justified to apply arbitrarily the thermal transpiration correction to the pressure across this boundary, because the thermal transpiration relation holds strictly only in the case of no net molecular flow across the boundary (in other words, to a gas at rest) (Ref. 2)*. It is therefore important to determine the ratio of the random molecular mass flux of the gas at rest to the net directed mass flux due to gas flow in our experiment.

The random molecular mass flux of a gas at rest is given by (Ref. 1)*

$$G = p \left(\frac{M}{2\pi RT} \right)^{1/2} \tag{3}$$

In the case of our experiment, the highest pressure is about 10^{-5} torr (1.3×10^{-2} dynes/cm^2). At this pressure, and at a temperature of 300 deg K, equation (3) gives $G = 1.9 \times 10^{-7}$ g sec^{-1} cm^{-2} (for oxygen).

The directed mass flux due to the net flow of gas in our experiment will now be calculated, using equation (1). From that equation, $\dot{m} = MQ/RT$. For a typical experiment in which the sample pressure is 10^{-5} torr, the flow rate, Q, is about 3×10^{-5} torr liters/sec. Using this value and a temperature of 300 deg K, $\dot{m} = 5 \times 10^{-8}$ g/sec. With a tube cross-sectional area of 3.8 cm^2, $G = \dot{m}/A = 1.3 \times 10^{-8}$ g sec^{-1} cm^{-2}. Thus, the ratio of random mass flux to directed mass flux is about 15. This ratio does not change appreciably with the pressure of the experiment as both fluxes are directly proportional to pressure level (assuming the sticking coefficient of the specimen remains constant). Thus, the random mass flux greatly predominates and therefore the thermal transpiration pressure correction should apply reasonably well.

* References are presented at the end of this Section 5-4.

The thermal transpiration relationship (Ref. 2) applied to our system gives

$$(p_A/p_B) = (T_1/T_2)^{1/2} \tag{4}$$

The following two equations can be written from the definition of conductance, C:

$$Q_1 = C_1\ (p_1 - p_A) \tag{5}$$

$$Q_2 = C_2\ (p_B - p_2) \tag{6}$$

Equations (4), (5), and (6) can be combined, eliminating p_A and p_B to give:

$$p_2 = \left(\frac{T_2}{T_1}\right)^{1/2} p_1 - \frac{Q_1}{C_1}\left(\frac{T_2}{T_1}\right)^{1/2} - \frac{Q_2}{C_2} \tag{7}$$

Equation (2) can be used to put Q_1 and Q_2 in terms of Q_o to yield

$$P_2 = \left(\frac{T_2}{T_1}\right)^{1/2} p_1 - \frac{Q_o(T_1\ T_2)^{1/2}}{C_1\ T_o} - \frac{Q_o\ T_2}{C_2\ T_o} \tag{8}$$

This equation gives the predicted pressure directly over the specimen as a function of the flow rate, temperatures, conductances, and p_1.

The conductance of a tube in molecular flow is given by the following equations (Ref. 3):

$$C_1 = 3.81\frac{D^3}{L_1}\left(\frac{T_1}{M}\right)^{1/2} \tag{9}$$

$$C_2 = 3.81\ \frac{D^3}{L_2}\left(\frac{T_2}{M}\right)^{1/2} \tag{10}$$

The internal diameter, D, measures 2.20 cm in both sections 1 and 2. Substituting this value, and M = 32 in (9) and (10) gives

$$C_1 = 7.18\ (T_1)^{1/2}/L_1 \qquad (11)$$

$$C_2 = 7.18\ (T_2)^{1/2}/L_2 \qquad (12)$$

Also, Q_o is given by

$$Q_o = 0.615\ (p_o - p_1) \qquad (13)$$

where the conductance (C_o) of 0.615 liters/sec has been measured previously and has been used to determine flow rates in prior experiments. If (11), (12), and (13) are substituted in (8), and if T_o is set equal to 295 deg K, there results

$$p_2 = \left(\frac{T_2}{T_1}\right)^{1/2} p_1 - \frac{\left(T_2\right)^{1/2} L}{3450} (p_o - p_1) \qquad (14)$$

Equations (8) and (14) can be considered the important equations: equation (8) being more general, and equation (14) applying specifically to the particular test apparatus.

Comparison of Test Data

The other prediction of the specimen pressure, p_2, can be made on the basis of the quartz gage which measures p_3. Using the thermal transpiration correction,

$$p_2 = p_3\ (T_2/T_3)^{1/2} \qquad (15)$$

where T_3 is approximately room temperature.

When the specimen pressure, p_2, was calculated from equations (14) and (15), using data from the 6th exposure, the comparison was still rather poor. The pressure predicted from equation (14) was higher than from (15) by a factor of almost 4 when the pressure was in the 10^{-5} range. It turns out that predicting the pressure from eqn. (14) involves taking a small difference between two relatively large numbers, which makes the pressure p_2 strongly dependent upon the constant which was calculated in (14) to be 3450. This constant of 3450 is in turn dependent upon physical measurements of the system, and upon the assumptions made and implied in the analysis. The constant of 3450 is therefore subject to error due to measurements (it involves, for example, the cube of tube diameter), and also error due to the assumptions.

If we now assume only that the form of eqn. (14) is correct, and that the constant is to be an empirically determined constant, we can determine a new value of the constant from the data of the 6th exposure. If the symbol K is substituted for the constant 3450 in (14) and the equation solved for K there results

$$K = \frac{(T_2)^{1/2} L(p_o - p_1)}{(T_2/T_1)^{1/2} p_1 - p_2} \tag{16}$$

The value for p_2 can be obtained from the measured value of p_3, with the thermal transpiration correction of eqn. (15).

Calculations for K for various test conditions of exposure No. 6 are given on the next page. Gage readings used are corrected for gage sensitivity for oxygen gas. A later gage calibration changed the sensitivities somewhat. However, the ratio of gage sensitivities had been accurately established, and each gage sensitivity changed only by a constant multiplier.

DETERMINATION OF K FROM EQUATION (16) (Exposure No. 6)

Date	8-12-64	8-13-64	8-17-64	8-18-64	8-19-64	8-21-64
Time	1:30 pm	1:10 pm	1:10 pm	1:33 pm	1:33 pm	8:10 am
pressure multiplier, λ	10^{-7}	10^{-6}	10^{-7}	10^{-6}	10^{-5}	10^{-5}
p_o indicated (torr)	6.4λ	6.0λ	5.3λ	8.6λ	5.7λ	6.0λ
p_1 indicated (torr)	1.7λ	1.2λ	1.75λ	1.6λ	1.05λ	1.1λ
p_3 indicated (torr)	0.78λ	0.24λ	0.86λ	0.24λ	0.11λ	0.13λ
p_o corrected (torr) (Note 1)	8.0λ	7.5λ	6.62λ	10.75λ	7.13λ	7.5λ
p_1 corrected (torr) (Note 1)	2.12λ	1.5λ	2.19λ	2.0λ	1.31λ	1.37λ
p_3 corrected (torr) (Note 2)	0.843λ	0.259λ	0.93λ	0.259λ	0.119λ	0.140λ
T_2 (deg.C)	980	980	1100	1100	1100	980
T_2 (deg.K)	1253	1253	1373	1373	1373	1253
T_1 (deg. K) (Assumed)	300	300	300	300	300	300
L (cm)	26.4	26.4	26.4	26.4	26.4	26.4
p_2 (calculated from Eq. (15))	1.72λ	0.528λ	1.99λ	0.554λ	0.255λ	0.286λ
K (calculated from Eq. (16))	2110	2220	1610	2290	2230	2280

(Note 1): Gage sensitivity used = 8.0 per torr.

(Note 2): Gage sensitivity used = 9.3 per torr.

It is apparent from equation (16) that a constant multiplier applied to each gage reading will not change the value of K.

The readings selected for entry in the table were at the end of each phase of the 6th exposure, so that the greatest stability would have been achieved for each reading.

The values of K are quite close except for the 3rd column. A check of the strip-chart record for the data of the 3rd column, indicated that the "pump gage" (p_o) tended to drift upward for a period of time after the gage panel was balanced for the reading which was entered in the table. This drift is unexplained, and did not occur during the other phases of the 6th exposure. It is felt that the data of the 3rd column should be disregarded in establishing the value of K. An arithmetic average of the remaining values for K gives:

$$K = 2226$$

This value of K is 65% of the calculated value of 3450 in equation (14).

Conclusions

An equation has been derived which can be used to predict the pressure over the specimen from knowledge of the temperatures and the pressures of the "pump" and "sample" gages. The equation is obtained by substituting an empirically-derived constant in equation (14). The final equation, based upon the data of the sixth exposure is:

$$p_2 = \left(\frac{T_2}{T_1}\right)^{1/2} p_1 - \frac{(T_2)^{1/2} L}{2226} (p_o - p_1) \qquad (17)$$

The pressure over the specimen can also be predicted by a thermal transpiration correction of the "quartz" gage reading (eqn. 15). The specimen pressure from equation (15) is perhaps the pressure which should be considered the "correct" specimen pressure, as it is obtained in a more straightforward manner than from equation (17).

The results of predicted pressures over the specimen (p_2) from equations (15) and (17) for the 6th exposure are summarized below (the data from the third column of the previous chart are omitted).

Date	P_1 Corrected (torr)	P_2 Predicted From Eqn. (15) (torr)	Eqn. (17) (torr)
8-12-64	2.12×10^{-7}	1.72×10^{-7}	1.85×10^{-7}
8-13-64	1.5×10^{-6}	5.3×10^{-7}	5.4×10^{-7}
8-18-64	2.0×10^{-6}	5.5×10^{-7}	4.4×10^{-7}
8-19-64	1.31×10^{-5}	2.5×10^{-6}	2.5×10^{-6}
8-21-64	1.37×10^{-5}	2.8×10^{-6}	2.3×10^{-6}

The maximum deviation is 20% of the equation (15) pressure.

C. A. Barrett of the Lewis Research Center has computed values for "K" from later exposures.

References for Section 5-4:

1. S. Dushman and J.M.Lafferty, "Scientific Foundations of Vacuum Technique", Second Edition, John Wiley and Sons, 1962, p. 14.

2. *Ibid*., p.58.

3. *Ibid*., p. 88.

5-5. Calibration Data for Secondary Standard Gages

The final calibration of the "secondary standard" gages was performed by Varian Associates, Palo Alto, California. Their original data are presented in this Section.

The data are presented first for gage tube number MG-10 (General Electric Model 22GT102) with its control panel (General Electric Model 22GC101, Serial 40002). The second set of data is for gage tube number MG-11 (General Electric Model 22GT102) with its control panel (General Electric Model 22GC101, Serial 30013). For each gage, calibrations were performed for both oxygen and nitrogen.

VARIAN associates

611 HANSEN WAY • PALO ALTO, CALIFORNIA • 326-4000

IONIZATION GAUGE CALIBRATION

This folder contains the pressure calibration data for a hot cathode ionization gauge with its control unit. This gauge was calibrated using an accurately known pumping speed (s) with a measurement of flow rate (q) to establish a calibrated pressure $P = q/s$. The maximum error of this technique with the equipment used is $\pm$ 6%. For a detailed description of this calibration method, refer to the paper by W. H. Hayward and R.L. Jepsen, "A Simple High Vacuum Gauge Calibration System", Transactions of the American Vacuum Society; 1962, page 459.

Pertinent Data

Calibration Date 18-23 DEC 1964

Gauge Type and Number G.E. MOD. № 22GT102 № MG-10 (DESIGNATED "V1" BY G.E.)

Control Unit Type and Number G.E. Mod. 22GC101 S.N. 40002

Test Gas NITROGEN AND OXYGEN

Emission Current 0.1 ma.

Degas Method and Power Run emission current at 40 ma.

Plot of P_a/I_+ vs P_a is enclosed.

Definitions

I_+ - the gauge indication as read on the electrometer.
Pa - absolute (calibrated) pressure.
Pa/I_+ - correction factor.

To find the absolute pressure, multiply the gauge pressure indication I_+ by the appropriate correction factor as determined from the plot

Pa/I_+ vs P_a

Degassing Procedure

The degassing and bakeout were as outlined in the round robin instructions.

Calibration Done By: [signature]
Maurice Carlson, Engineer

Approved by: [signature] ABF
Arthur B. Francis, Director
Vacuum Applications Laboratory
Vacuum Products Division
Varian Associates

Data Sheet For Gauge No. MG-10 NITROGEN

I_g (amps)		P_a (torr)		P_a/I_g
Indication	Scale	Indication	Scale	
6.18	10^{-10}	5.58	10^{-7}	903
9.35	-10	9.37	-7	1002
0.98	-9	9.37	-7	956
2.53	-9	2.82	-6	1115
4.71	-9	5.42	-6	1151
7.85	-9	8.74	-6	1113
10.00	-9	1.08	-5	1080
1.10	-8	1.08	-5	982
9.45	-8	1.18	-4	1249
4.00	-8	4.86	-5	1215
6.32	-8	7.67	-5	1214
2.35	-8	2.83	-5	1204
2.90	-8	3.49	-5	1203
1.68	-8	1.89	-5	1125

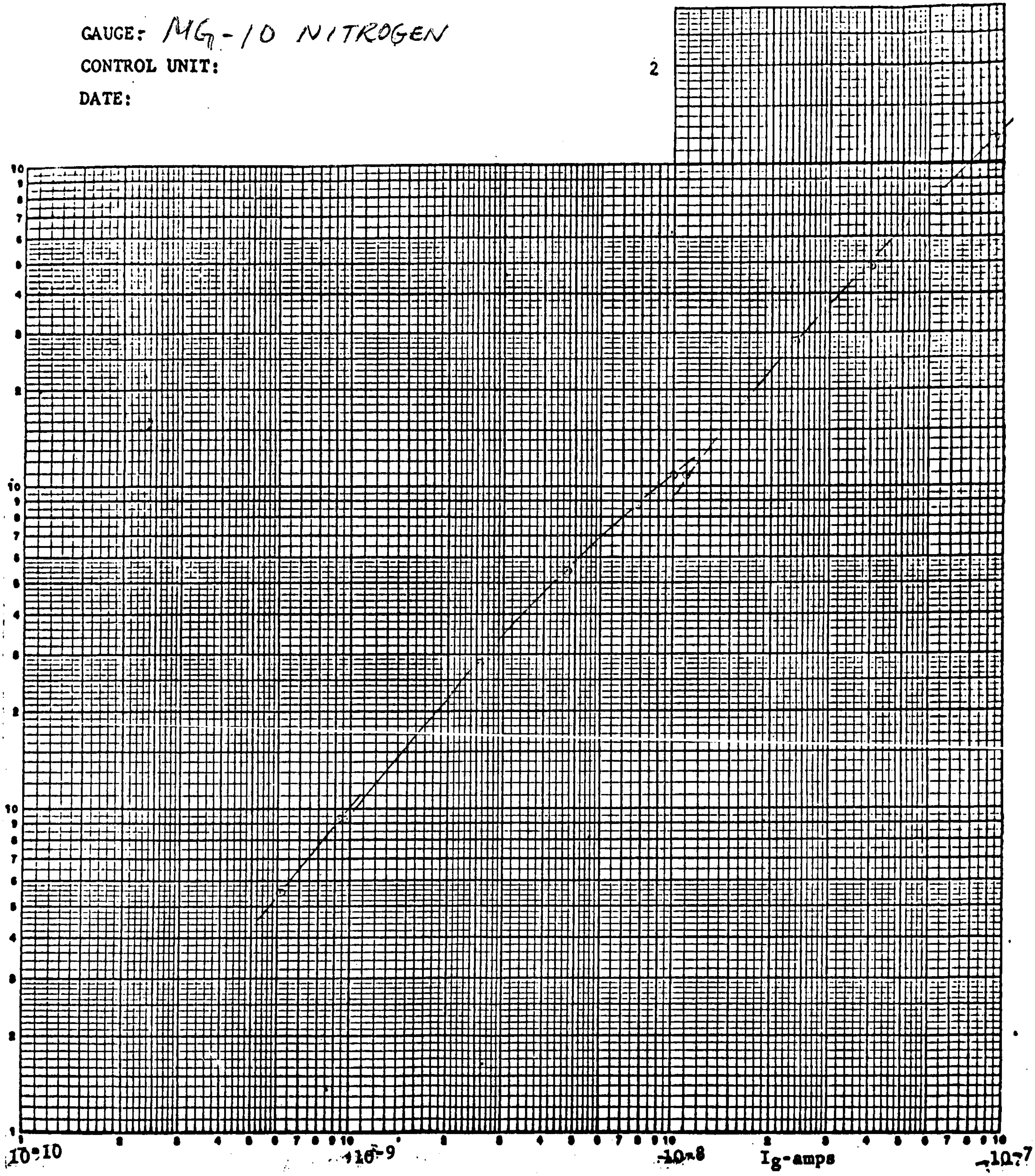
Additional Information:

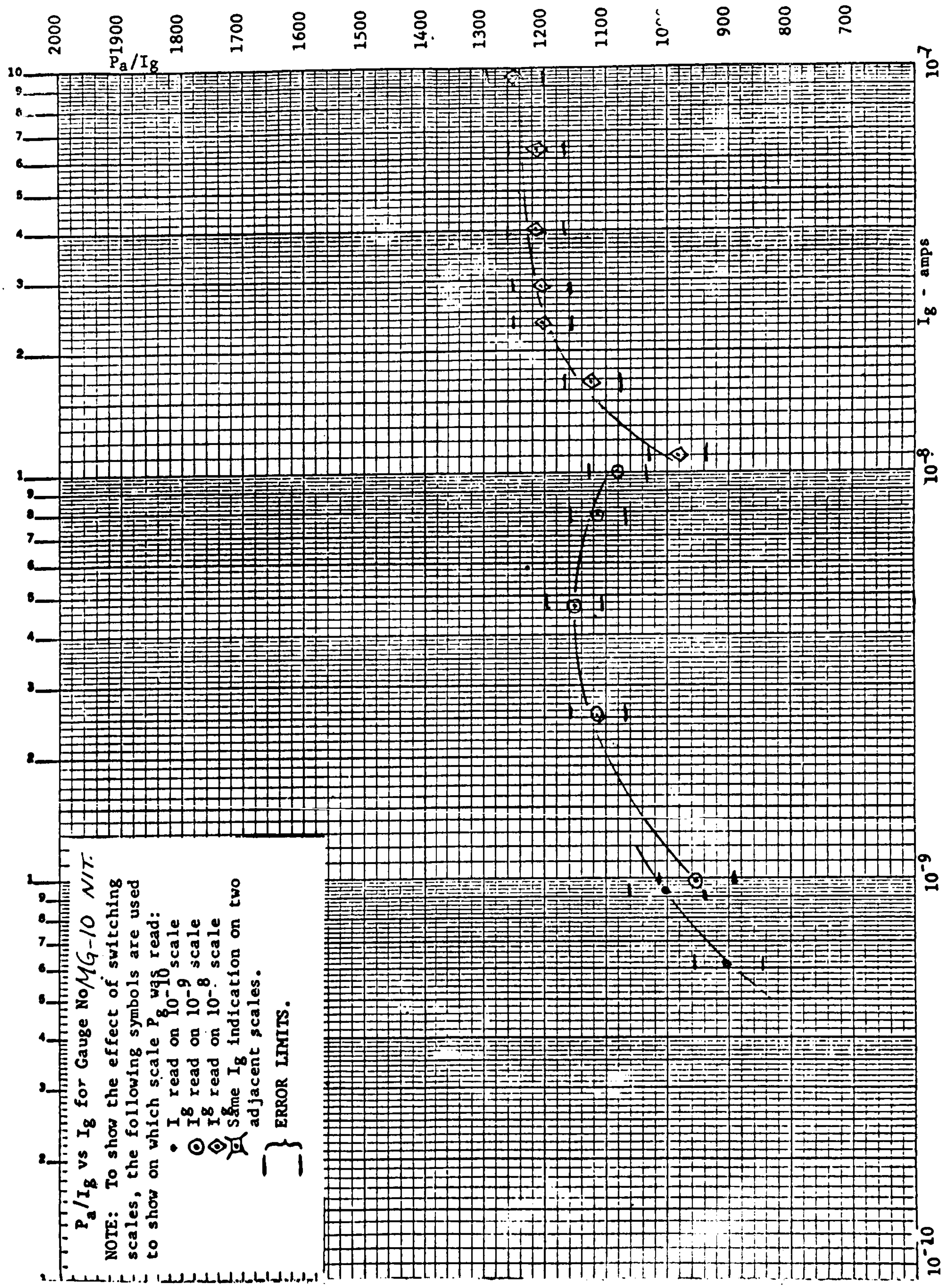
1. All values are for 20°C
2. TUBE WAS ORIENTED WITH AXIS OF GRID CAGE HORIZONTAL AND GE LABEL UPPERMOST.
3. THORIATED FILAMENT WAS USED.

GAUGE: MG-10 NITROGEN

CONTROL UNIT:

DATE:




Pa/Ig vs Ig for Gauge No MG-10 NIT.
NOTE: To show the effect of switching scales, the following symbols are used to show on which scale Pg was read:
• Ig read on 10^-10 scale
Ig read on 10^-9 scale
Ig read on 10^-8 scale
Same Ig indication on two adjacent scales.
ERROR LIMITS.
Pa/Ig
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
900
800
700
Ig - amps
10^-7
10^-8
10^-9
10^-10

Data Sheet For Gauge No. MG-10 OXYGEN

I_g (amps)		P_a (torr)		P_a/I_g
Indication	Scale	Indication	Scale	
6.05	10^{-10}	7.74	10^{-7}	1279
9.78	-10	1.29	-6	1319
0.96	-9	1.29	-6	1343
2.50	-9	3.64	-6	1456
4.43	-9	6.56	-6	1481
6.85	-9	1.01	-5	1474
10.00	-9	1.46	-5	1460
0.96	-8	1.46	-5	1520
2.52	-8	3.83	-5	1520
4.97	-8	7.26	-5	1461
7.96	-8	1.12	-4	1407
9.65	-8	1.37	-4	1420

Additional Information:

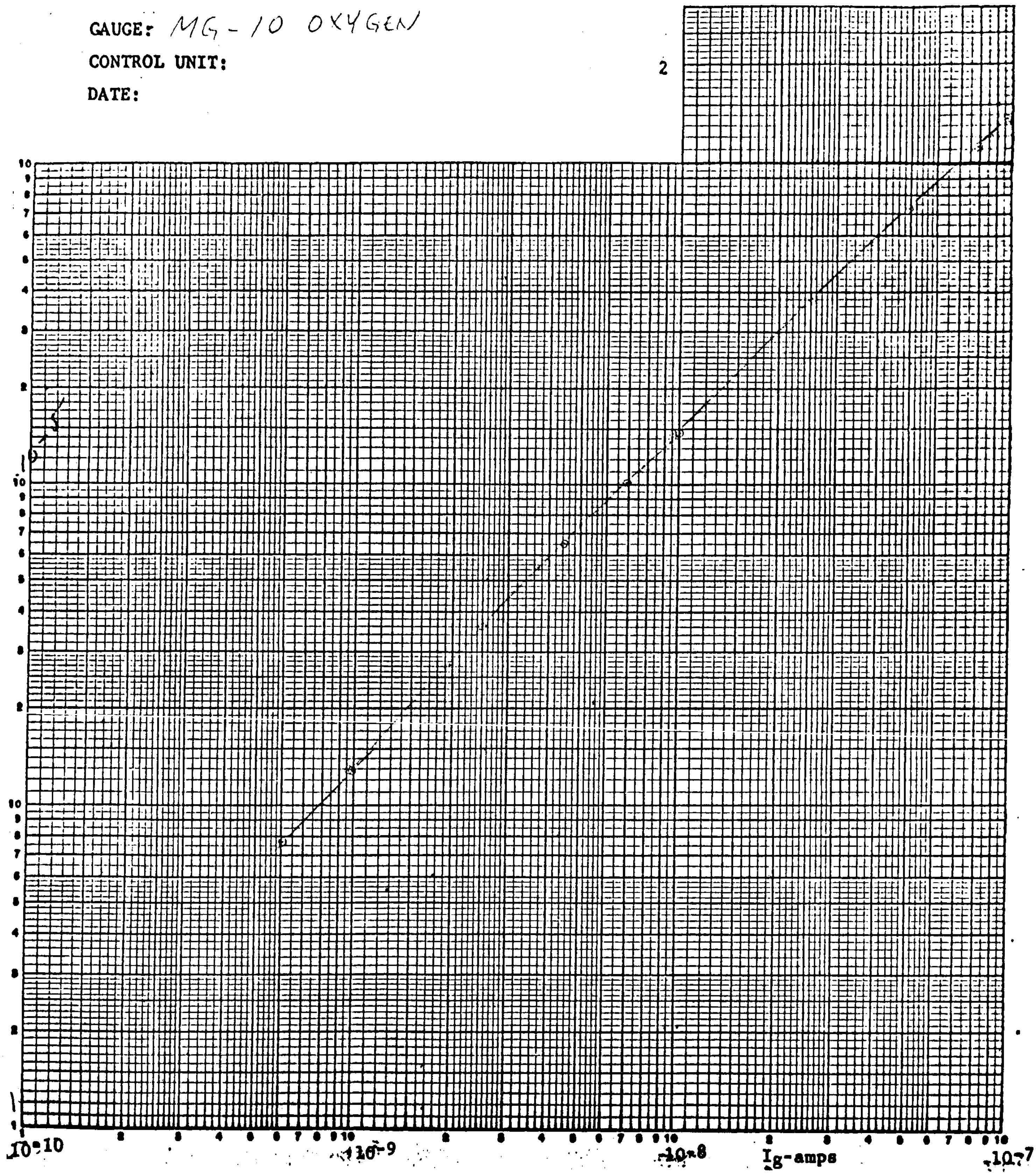
1. All values are for 20°C
2. TUBE WAS ORIENTED WITH AXIS OF GRID CAGE HORIZONTAL AND GE LABEL UPPERMOST.
3. THORIATED FILAMENT WAS USED.

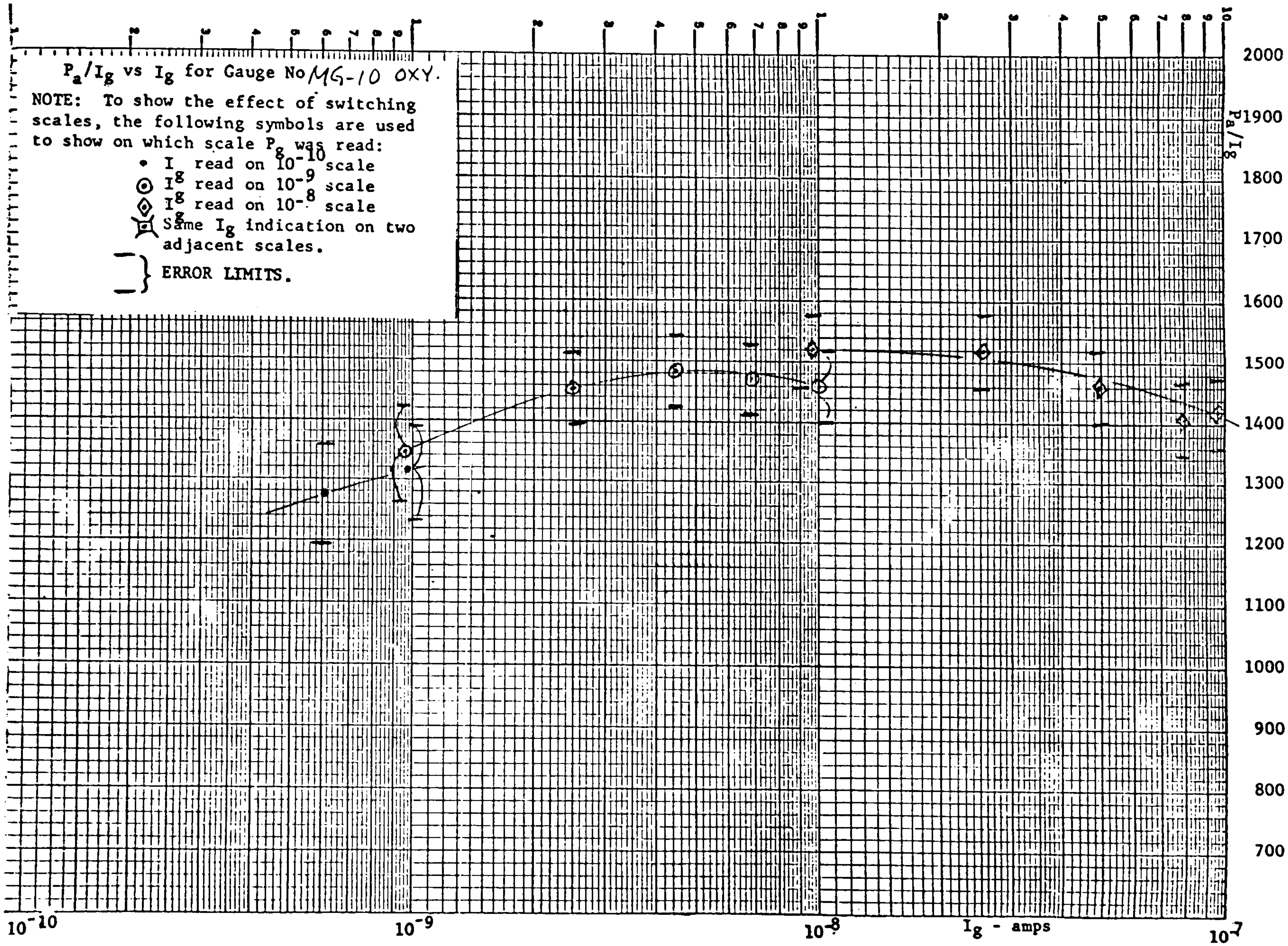
I

GAUGE: MG-10 OXYGEN

CONTROL UNIT:

DATE:





P_a/I_g vs I_g for Gauge No MG-10 OXY.
NOTE: To show the effect of switching scales, the following symbols are used to show on which scale P_g was read:
• I_g read on 10^{-10} scale
I_g read on 10^{-9} scale
I_g read on 10^{-8} scale
Same I_g indication on two adjacent scales.
ERROR LIMITS.
P_a/I_g
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
10^{-10}
10^{-9}
10^{-8}
10^{-7}
I_g - amps

VARIAN associates

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IONIZATION GAUGE CALIBRATION

This folder contains the pressure calibration data for a hot cathode ionization gauge with its control unit. This gauge was calibrated using an accurately known pumping speed (s) with a measurement of flow rate (q) to establish a calibrated pressure $P = q/s$. The maximum error of this technique with the equipment used is $\pm$ 6%. For a detailed description of this calibration method, refer to the paper by W. H. Hayward and R.L. Jepsen, "A Simple High Vacuum Gauge Calibration System", Transactions of the American Vacuum Society; 1962, page 459.

Pertinent Data

Calibration Date 18-23 DEC 1964

Gauge Type and Number G.E. Mod No 22GT102 No MG-11

Control Unit Type and Number GE, Mod 22GC101 S.N. 30013

Test Gas NITROGEN AND OXYGEN

Emission Current 0.1 ma.

Degas Method and Power Run Emission at 40 ma

Plot of P_a/I_+ vs P_a is enclosed.

Definitions

I_+ - the gauge indication as read on the electrometer.
Pa - absolute (calibrated) pressure.
Pa/I_+ - correction factor.

To find the absolute pressure, multiply the gauge pressure indication I_+ by the appropriate correction factor as determined from the plot

Pa/I_+ vs P_a

Degassing Procedure

The degassing and bakeout were as outlined in the round robin instructions.

Calibration Done By: M Carlson
Maurice Carlson, Engineer

Approved by: Wayne E. Phillips for ABF
Arthur B. Francis, Director
Vacuum Applications Laboratory
Vacuum Products Division
Varian Associates

Data Sheet For Gauge No. MG-11 NITROGEN

I_g (amps)		P_a (torr)		P_a/I_g
Indication	Scale	Indication	Scale	
4.96	10^{-10}	5.58	10^{-7}	1125
7.33	-10	9.37	-7	1278
0.79	-9	9.37	-7	1186
2.13	-9	2.82	-6	1324
3.99	-9	5.42	-6	1362
6.50	-9	8.74	-6	1345
8.15	-9	1.08	-5	1325
0.85	-8	1.08	-5	1271
7.75	-8	1.18	-4	1523
9.03	-8	1.41	-4	1561
3.20	-8	4.86	-5	1519
5.04	-8	7.67	-5	1522
1.94	-8	2.83	-5	1459
2.35	-8	3.49	-5	1485
1.36	-8	1.89	-5	1390

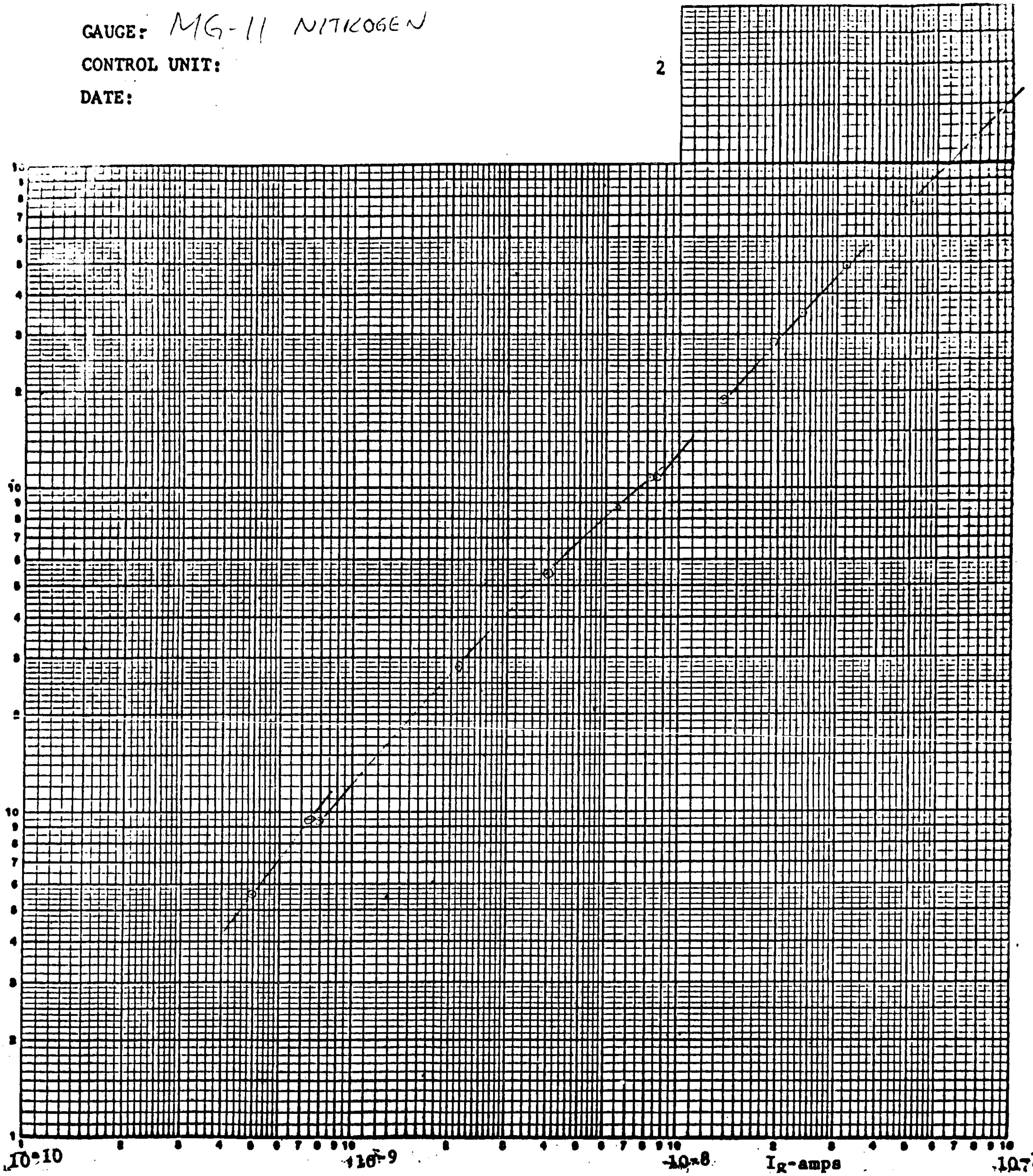
Additional Information:

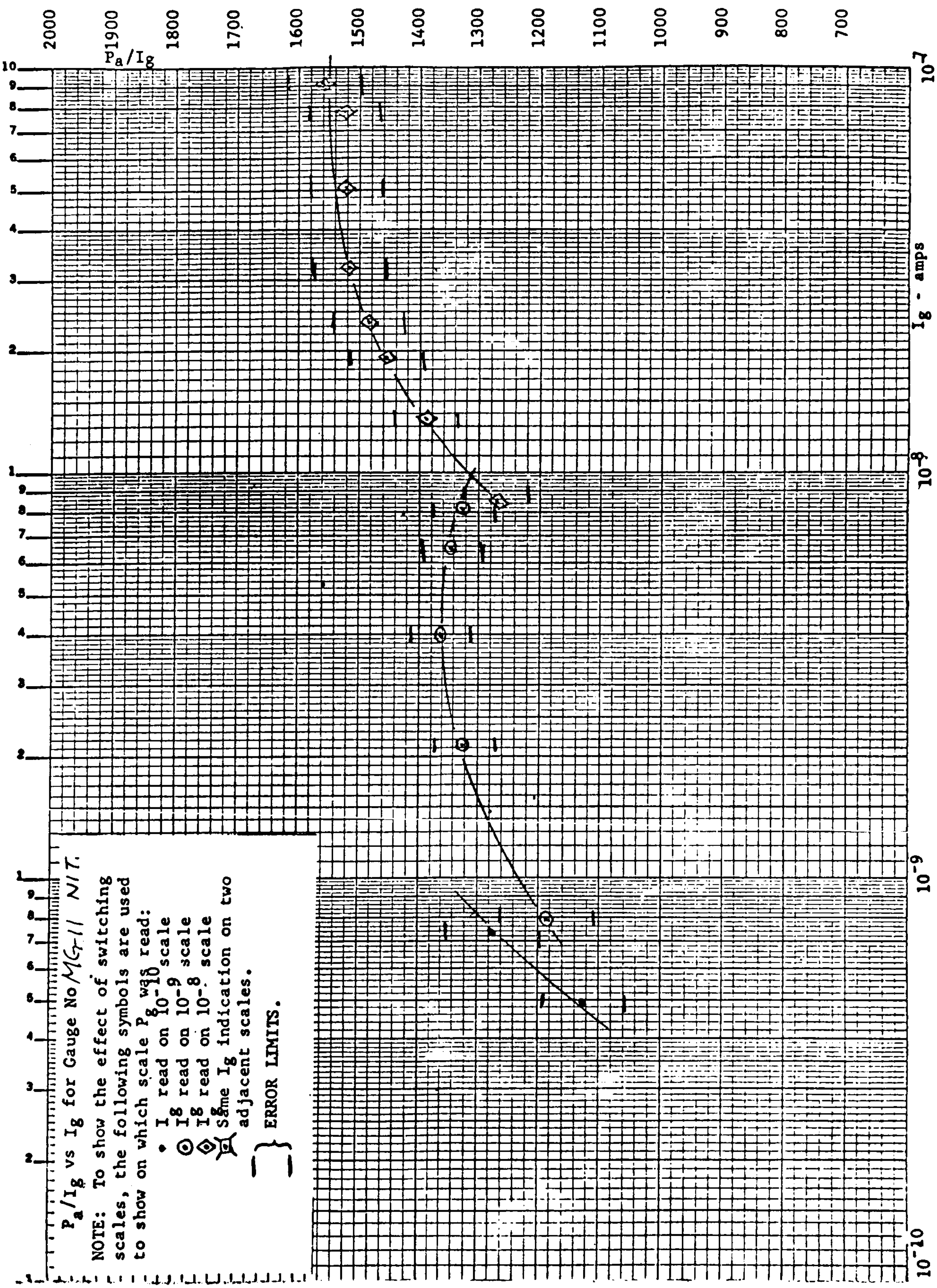
1. All values are for 20°C
2. TUBE WAS ORIENTED WITH AXIS OF GRID CAGE HORIZONTAL AND GE LABEL UPPERMOST
3. THORIATED FILAMENT WAS USED.

GAUGE: MG-11 NITROGEN

CONTROL UNIT:

DATE:





Pa/Ig
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
10^{-7}
10^{-8}
10^{-9}
10^{-10}
Ig - amps
P_a/I_g vs I_g for Gauge No MG-11 NIT.
NOTE: To show the effect of switching scales, the following symbols are used to show on which scale P_g was read:
• I_g read on 10^{-10} scale
⊙ I_g read on 10^{-9} scale
◇ I_g read on 10^{-8} scale
Same I_g indication on two adjacent scales.
ERROR LIMITS.

Data Sheet For Gauge No. MG-11 OXYGEN

I_g (amps)		P_a (torr)		P_a/I_g
Indication	Scale	Indication	Scale	
5.14	10^{-10}	7.74	10^{-7}	1506
7.90	-10	1.29	-6	1633
0.82	-9	1.29	-6	1573
2.20	-9	3.64	-6	1655
3.81	-9	6.56	-6	1722
5.80	-9	1.01	-5	1744
8.16	-9	1.46	-5	1789
0.81	-8	1.46	-5	1802
2.14	-8	3.83	-5	1790
4.04	-8	7.26	-5	1797
6.35	-8	1.12	-4	1764
7.61	-8	1.37	-4	1800
9.62	-8	1.76	-4	1830

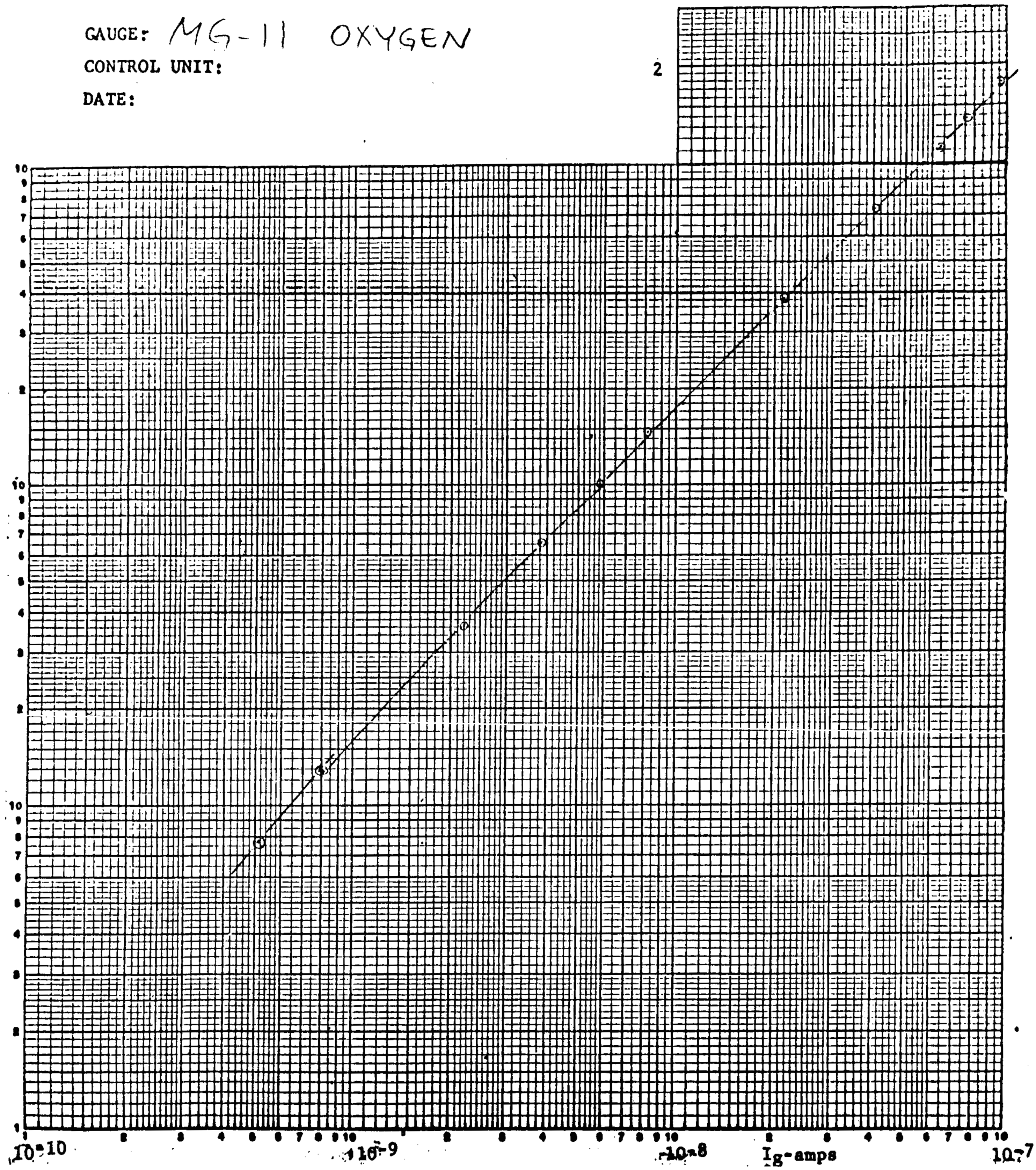
Additional Information:

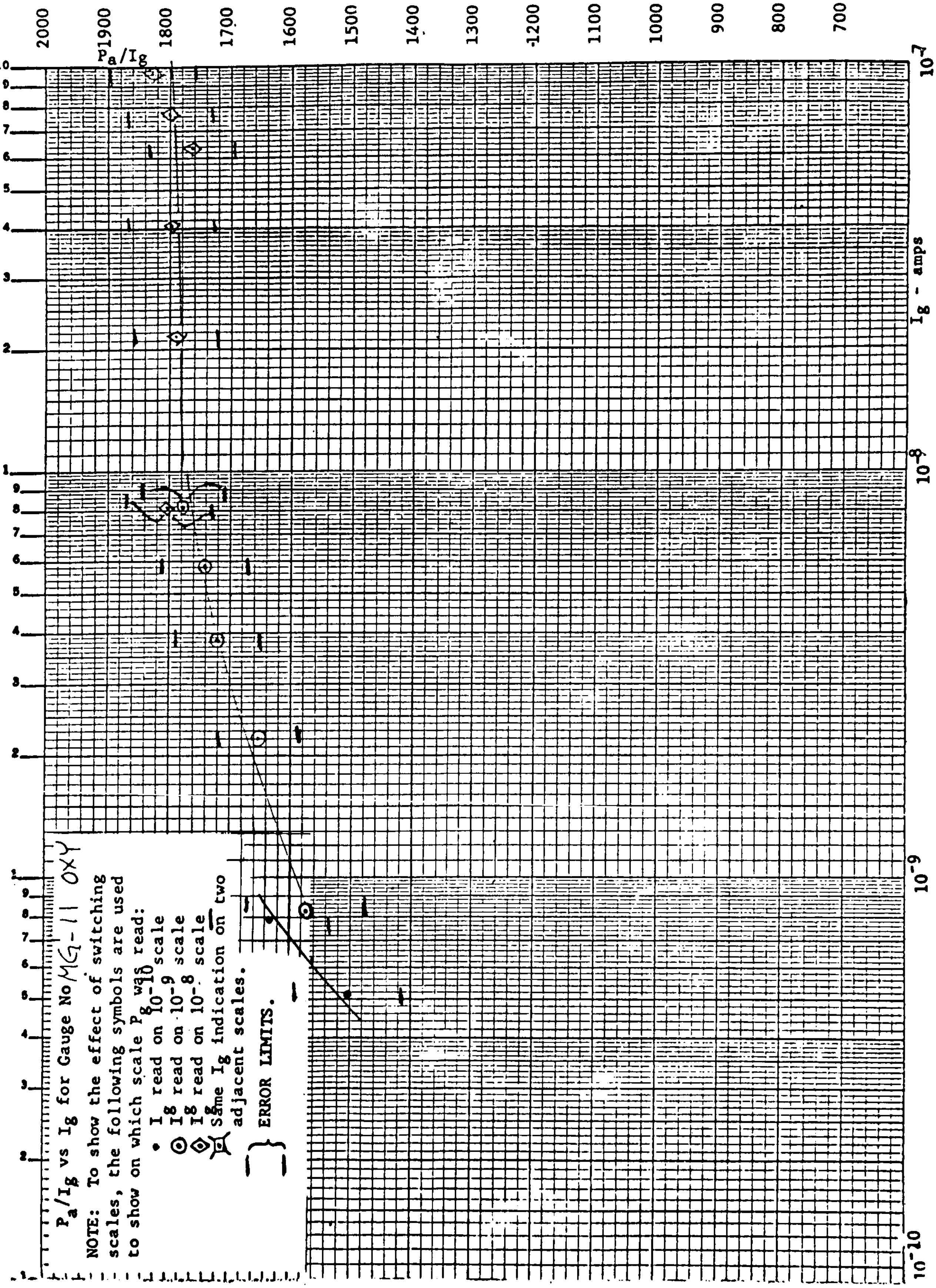
1. All values are @ 20°C
2. GAGE WAS ORIENTED WITH AXIS OF GRID CAGE HORIZONTAL AND G.E. LABEL UPPERMOST
3. THORIATED FILAMENT WAS USED.

GAUGE: MG-11 OXYGEN

CONTROL UNIT:

DATE:





Pa/Ig
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
10^-7
10^-8
10^-9
10^-10
Ig - amps
Pa/Ig vs Ig for Gauge No MG-11 OXY
NOTE: To show the effect of switching scales, the following symbols are used to show on which scale Pg was read:
• Ig read on 10^-10 scale
⊙ Ig read on 10^-9 scale
◇ Ig read on 10^-8 scale
Same Ig indication on two adjacent scales.
ERROR LIMITS.

5-6. Calibration of System Gages Against Secondary Standards

After the calibration of the secondary standard gages by Varian (Section 5-5), the three system gages and their respective control panels were calibrated against the secondary standards. This was done essentially in five steps.

Step 1.

Two of system gages (the "pump" gage and the "quartz" gage) were mounted in a cluster with the two secondary standard gages. Figure 17 is a photo of this gage cluster, and Figure 18 shows the instrumentation for the secondary standard gages. The "sample" gage was not mounted in the same cluster because of lack of room, and because a previous calibration related the "sample" gage readings to the other two system gages.

The oxygen pressure was brought slowly from the mid-10^{-8} torr to the mid-10^{-5} torr range, and readings were taken periodically on all four gages. The "true" pressure at each point was taken as the arithmetic average of the corrected pressure of the two secondary standard gages.

Unfortunately, the Varian calibration of the two secondary standard gages extended down only to the high 10^{-7} torr range. In order to extend the calibration to lower pressures, the following two steps were necessary.

Step 2.

The control panels of the two secondary standard gages were calibrated for collector current and emission current, corresponding to the entire pressure range, thereby establishing the control panel correction factors, F_c and F_e, of Section 3.

Step 3.

Knowledge of the correction factors from Step 2 permitted calculation of the gage sensitivities of the two secondary standard gages, from Equation (3)

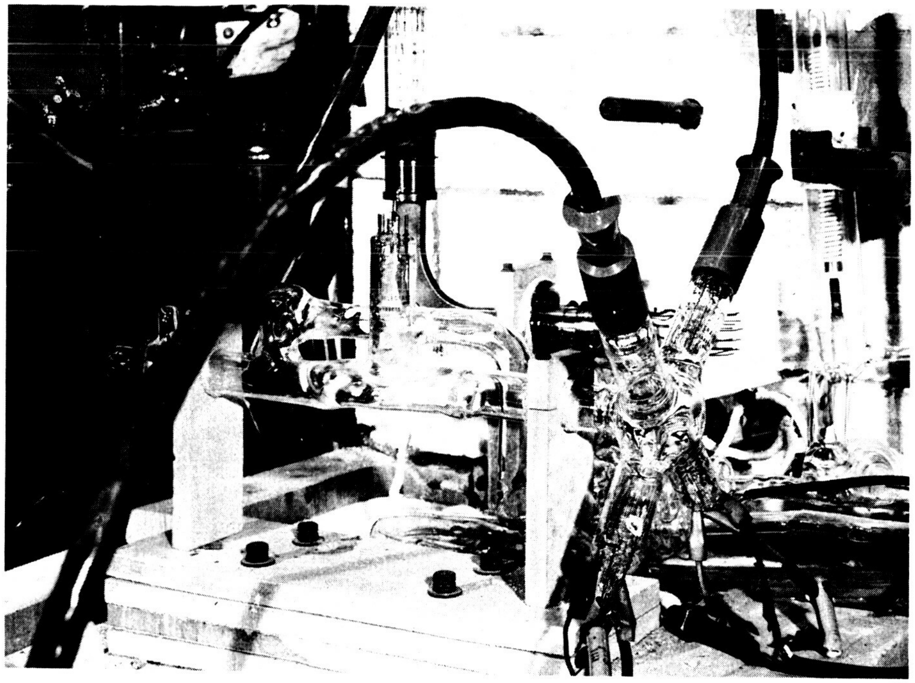


FIGURE 17. GAGE CALIBRATION

(ATL Photo 845602)
The two "secondary standard" gages are mounted at the top, and the "pump" and "quartz" gages from the system are at the bottom.

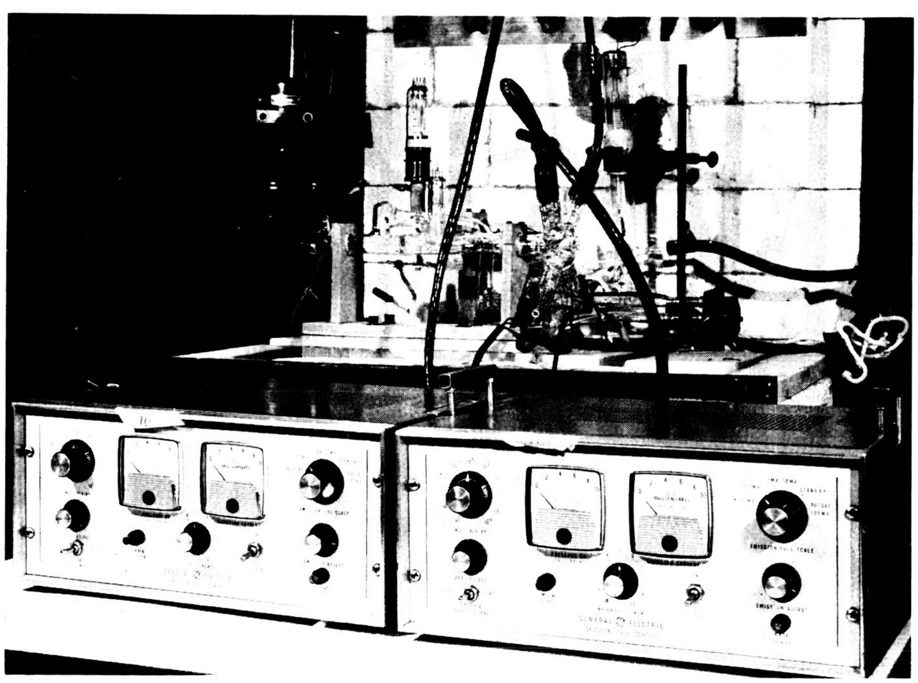


FIGURE 18. GAGE INSTRUMENTATION

(ATL Photo 845601)
The control panels for the secondary standard gages are shown in the foreground.

of Section 3. The 10 values of sensitivity thus found for each gage were averaged to give

For Gage MG 10, s = 7.09 per torr

For Gage MG 11, s = 5.68 per torr

The standard deviations in the values of sensitivity from which these averages were taken were 0.16 and 0.13, respectively.

Step 4.

With the gage tube sensitivities established for the secondary standards, and the control panel corrections known, the "true" pressures were calculated in the low pressure range from eqn. (3) of Section 3, with the assumption that the gage tube sensitivities of the secondary standards remained constant at the lower pressures. Thus, the calibration of the two system gages were extended to pressures below the Varian calibration.

Step 5.

The calibration curve for the "sample" ion gage was derived from a calibration made after the sixth exposure, in which all three system gages were mounted in a cluster and the readings checked in a manner similar to Step 1. Figure 19 is a plot of the results. In all three decades of interest, the ratio of "quartz" gage readings to "sample" gage readings was 1.20, within an accuracy of $\pm$ 3% except for 3 readings out of the 23 taken. The uncorrected control panel readings for the "quartz" gage were therefore calculated to be 1.20 times the uncorrected readings for the "sample" gage.

Final Calibration Plots

The final calibrations are plotted in Figures 20, 21, 22, and 23, on the following pages.

FIGURE 19

PRESSURE MEASUREMENTS OBTAINED FROM THREE ION GAGE TUBES MOUNTED CLOSE TOGETHER

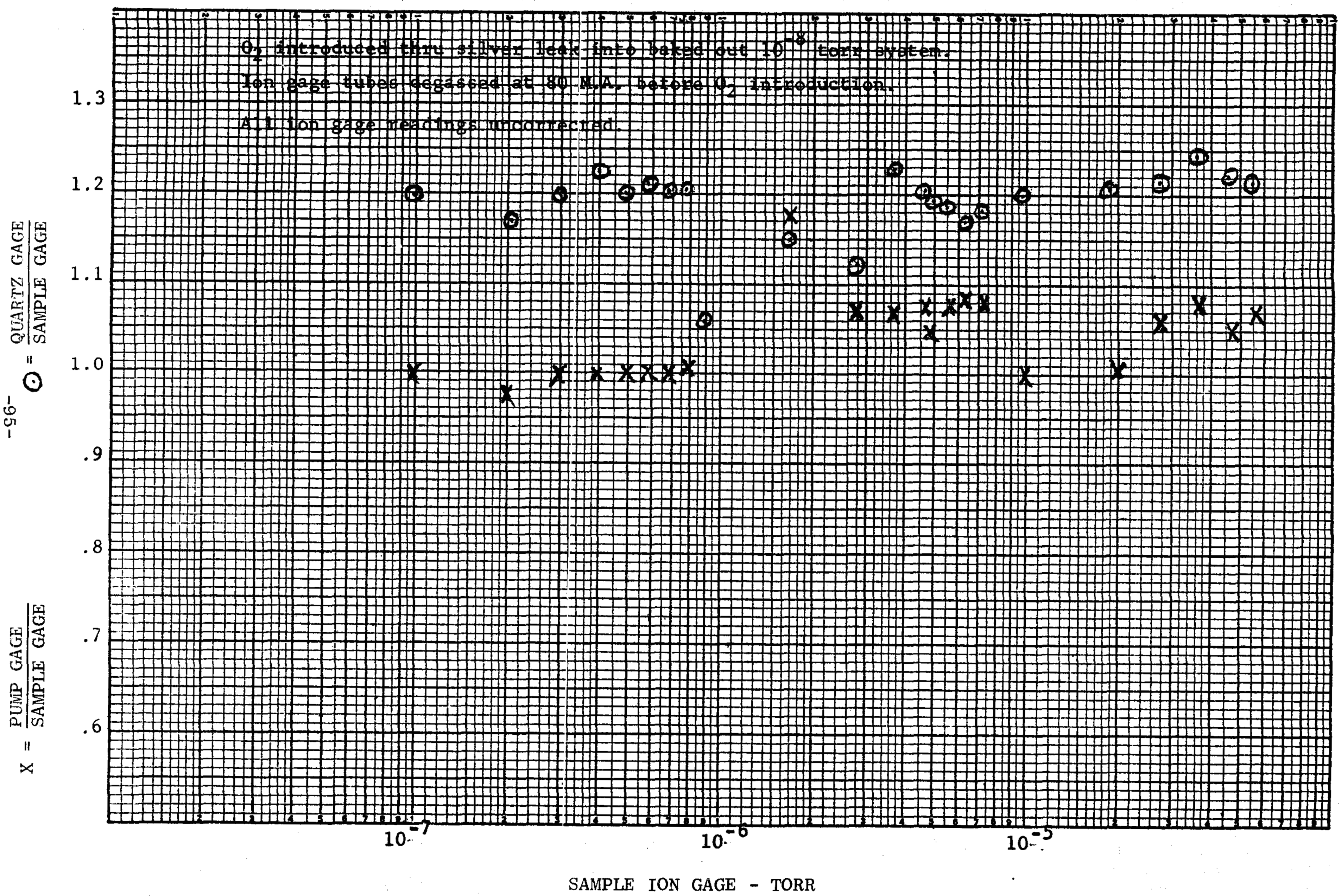


FIGURE 20

CALIBRATION CURVE FOR

OXYGEN GAS

(10^{-5}) TORR PRESSURE RANGE

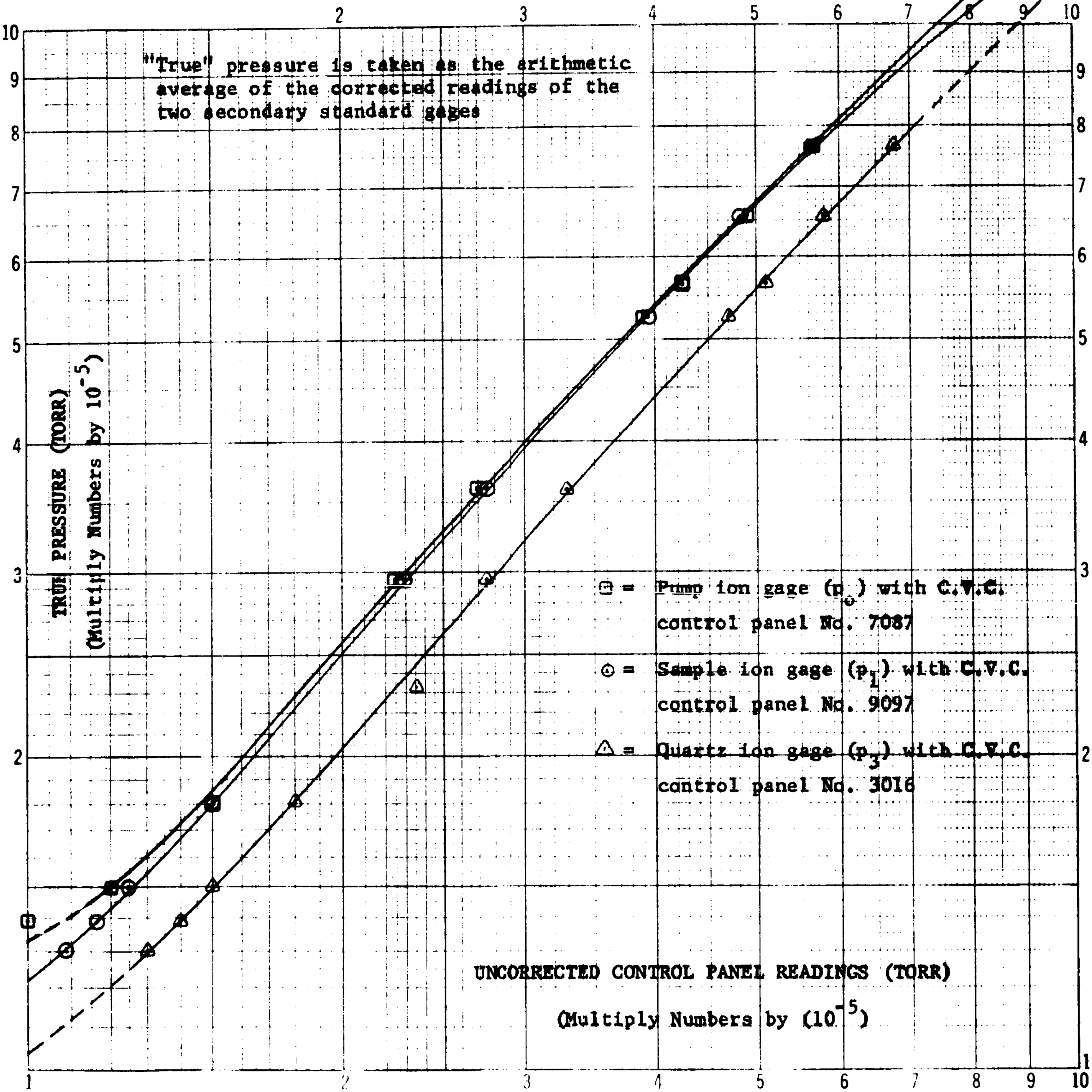


FIGURE 21

CALIBRATION CURVE FOR

OXYGEN GAS

(10^{-6}) TORR PRESSURE RANGE

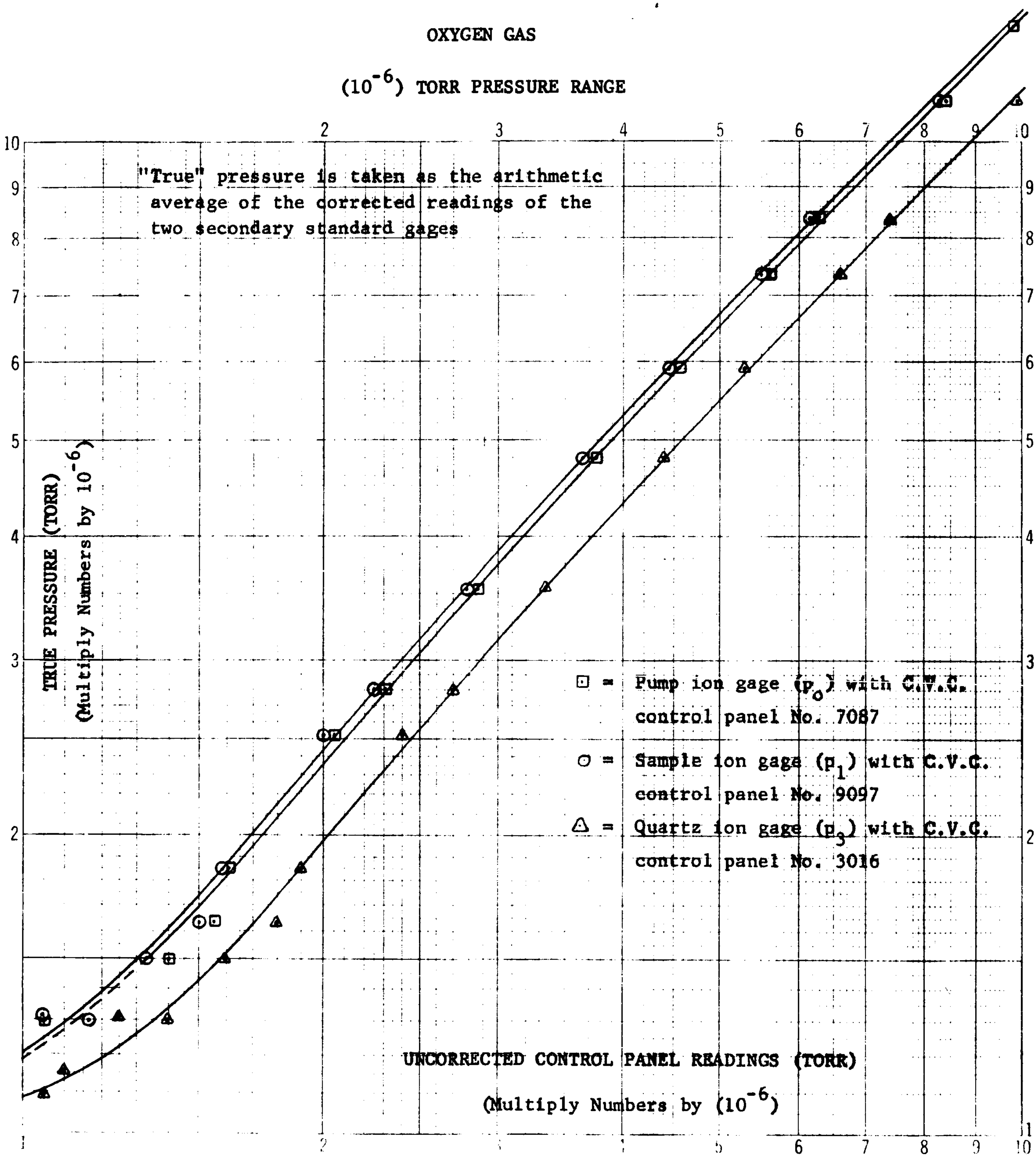


FIGURE 22

CALIBRATION CURVE FOR

OXYGEN GAS

(10^{-7}) TORR PRESSURE RANGE

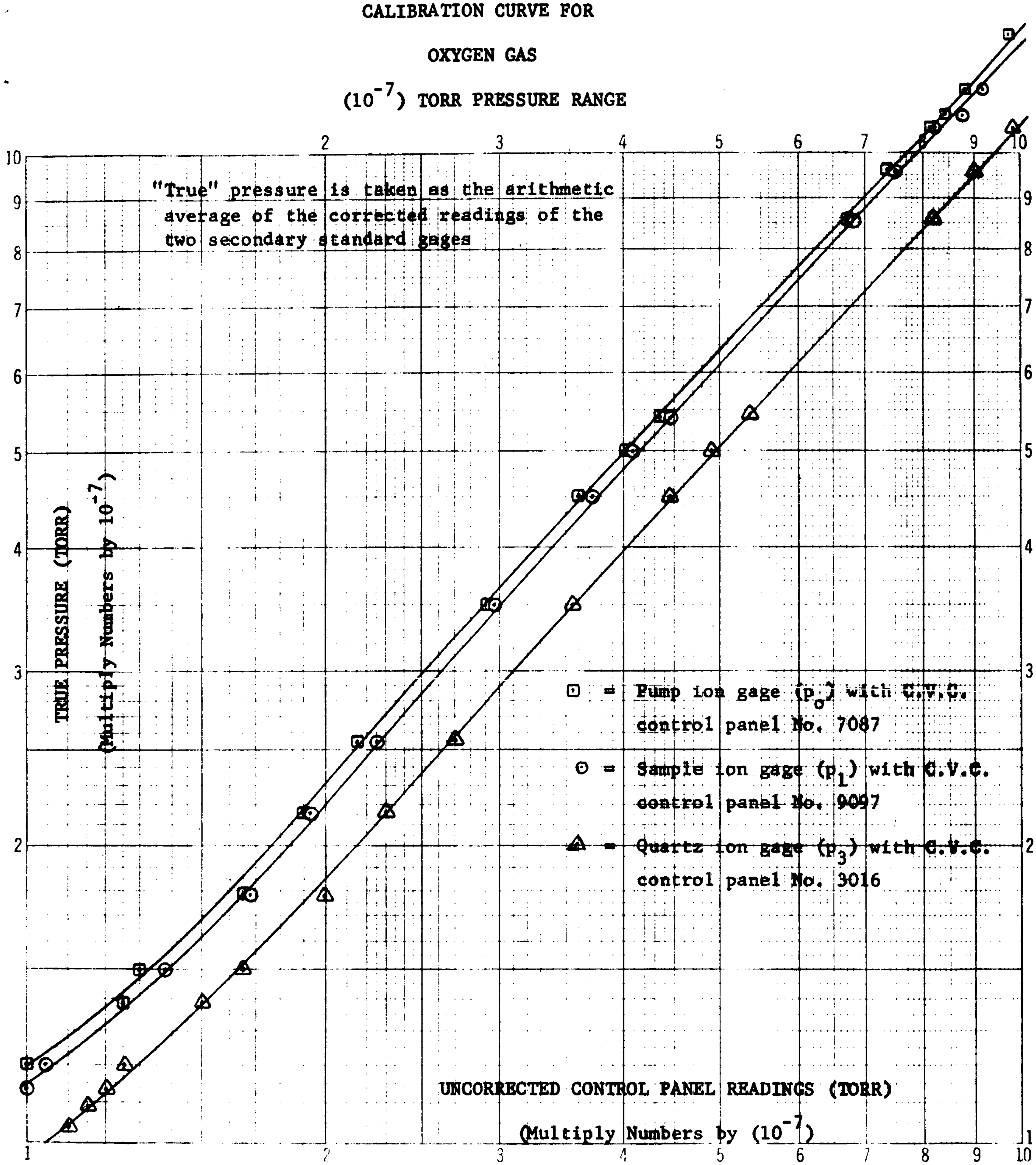


FIGURE 23

CALIBRATION CURVE FOR

OXYGEN GAS

(10^{-8}) TORR PRESSURE RANGE